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Analysis of Gas Plant Sludges for Cyanide and Total Elemental Contents Results of an Interlaboratory Study

Analysis of Gas Plant Sludges for Cyanide and Total Elemental Contents
- Results of an Interlaboratory Study

By

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EXECUTIVE SUMMARY

This report presents the results of an interlaboratory study for the analysis of cyanide and the elements Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se Tl, V, Zn in gas plant sludges.

Five standard and six sludge samples were submitted for analysis by the participating laboratories. The standard samples were made of one ICP aqueous sample, two digested certified standards, and two certified standards. The six sludges were submitted from CPA members' gas plants in the province.

The analysis of the submitted samples for the requested elements varied widely between labs, with responses from 20 to 90% of the requested analyses performed. This wide variability did not produce a database of elemental concentration results suitable for statistical analysis. The data provided was thus analyzed using recoveries of elements from the standards (using control charts), and comparison of the variability of the sludge results.

The recovery of the elements seems to be best for the ICP standard, followed by the digestate and the certified standard samples. The recovery of the elements varied from acceptable for most labs to almost no recovery in some cases. Differences in methodologies did not seem to cause any apparent changes in recoveries for these standards. The sludge results showed wide variability between labs (CV of 10 to 200%), which was exacerbated by the low number of samples analyzed and/or the low concentrations of the elements. Several of the elements showed good recovery, especially those that had many results reported. Differences in methodologies were not observed in the analysis of the sludges.

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1. INTRODUCTION

The Canadian Petroleum Association (CPA) published a report (CPA, 1987) summarizing the results of analysis of several sludges for organic and inorganic parameters. This report indicated poor results and inconsistencies found with the methods used to analyze for cyanide and total contents of several trace elements.

After discussions between the Canadian Petroleum Association Sludge Study Review Committee (CPASSRC) and the Alberta Research Council (ARC), a proposal for a jointly funded project was submitted by ARC and accepted by CPA that addressed the above noted results and inconsistencies reported in the 1987 CPA report. The proposal called for a survey of the pertinent literature and for a workshop on heavy metal analysis of gas plant sludges.

A literature search of the methods used for the total elemental analysis of waste sludges was conducted and included other materials such as sediments and soils. The databases searched were Chemical Abstracts, AGRICOLA and ENVIROLINE. The methods found in the literature search were summarized and presented in tables where the literature references were listed by waste type (or other), method used, results, and advantages/disadvantages. Based on the literature search, ARC suggested several methods appropriate for the total elemental analysis of the gas plant sludges. The listing of methods and ARC's 'suggested' method(s) were used as a basis for focused discussion and method selection for testing purposes in the workshop on heavy metal analysis in gas plant sludges.

The workshop was organized on November 23rd, 1987 and attended by members of the CPASSRC, qualified chemists in some of the private and government laboratories in Alberta, and ARC. The participants considered ARC proposed methods which were based on the literature search conducted previously. The result of the discussions in this meeting were summarized by ARC and distributed to all invited scientists including the set of methods (to be tested) commented on, discussed, and recommended by the meeting participants.

Following the workshop recommendations as to the "appropriate" methods to be tested by ARC laboratory, ARC submitted a proposal (accepted for joint funding by the CPA) that addressed the methods to be tested for the total analysis of several trace elements and cyanide in gas plant sludges.

The tests for a method for the analysis of cyanides were conducted on three gas plant sludges, and the factors affecting the recovery of added cyanides were also studied. Two methods for cyanide analysis were tested and compared, these were the APHA method (APHA, 1980) recommended by the workshop participants, and the Knechtel and Conn (1979) procedure used in phase 2 of the CPA study. Three digestion methods and several acid treatment combinations were tested for the total elemental analysis of the three gas plant sludges and 2 composite standard samples.

The results of the above work were compiled in a report by Chow and Abboud (1989). The report presented total cyanide recoveries from the gas plant sludge samples and trace element contents of two certified standards and three gas plant sludges. The results of the analyses were also compared to assess the utility and appropriateness of the digestion method and acid treatments combinations. The report concluded that the most acceptable acid treatment was HNO_3 and HF acids followed by H_3BO_3 (except for B determination where H_3BO_3 is eliminated). A combination of either microwave or pressure cooker digestion with HNO_3, HF and H_3BO_3 was reported to offer the best quantitative elemental recoveries from reference standards. Open beaker digestion was shown to suffer from the loss of volatile elements through volatilization.

A workshop was held in November 1989 to discuss the results presented in Chow and Abboud (1989), and to plan for an interlaboratory study on gas plant sludge analysis. The summary of the discussions and recommendations are appended to this report as Appendix 2. Based on that workshop recommendation, ARC prepared a set of standard and gas plant sludge samples which were distributed to 11 participating laboratories. The results of this interlaboratory study will be presented in this report.

2. OBJECTIVES

The objective of this study is to test the performance of the gas plant sludge analysis methods (microwave or pressure cooker digestion) developed by Chow and Abboud (1989), when attempted in several participating laboratories. The participating laboratories were also encouraged to include analyses of the materials by their own methods so as to compare to the methods of Chow and Abboud (1989). This interlaboratory study will look for total metal contents and cyanide in certified standards and gas plant sludges. The list of the elements to be determined in the sludges are : Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se Tl, V, Zn and CN.

The methods were tested on 1 ICP standard solution, 2 digestate solutions (of standard samples), 2 standard samples that are similar to gas plant sludges, and on 6 gas plant sludge samples provided by the CPASSRC.

3. MATERIALS AND METHODS

3.1 MATERIALS

The materials tested were either gas plant sludges or certified standards. Six gas plant sludges (A-F) were used, and two composite standards were obtained by mixing NBS fly ash with NBS sediment (with and without organic or inorganic salt solutions of Hg, Sb, Se and Tl) or with CONOSTAN oil standards. Two digested standard samples, one ICP standard and one blank were also included.

The total number of samples prepared and sent for each laboratory was 56, which consisted of 42 sludge and 14 standard samples.

3.1.1 Standards

As was done for the previous phase of the work (Chow and Abboud, 1989), composite certified standards were used as recommended by the workshop participants. The standard samples sent out to the labs consisted of the following: ICP standard, blank, digestate 3, digestate 4, standard A and standard B.

The ICP standard consisted of a standard aqueous solution of 18 elements each at a concentration of approximately 10 mg/L.

Standard A was made up of a combination of NBS Fly Ash, NBS River Sediment, and Blended Standard S-21 (CONOSTAN, containing Sb) and the addition of Hg and Se organic standards and Tl aqueous standard (normally used for ICP calibration). Several of these standards were digested and the digestates combined and distributed as digestate 3.

Standard B was made of a combination of Blended Standards S-21 (CONOSTAN, containing Sb) and NBS Fly Ash samples, and the addition of Hg and Se organic standards and Tl aqueous standard (normally used for ICP calibration). Several of these standards were digested and the digestates combined and distributed as digestate 4.

Several blank samples (acid mixture only) were prepared during the digestion of standards 3 and 4, combined and then transferred to separate polypropolyne bottles.

Each of the participating laboratories were sent the following standard samples:

1. One 60 mL blank sample (acid mixture) in a polypropolyne bottle.

2. One ICP aqueous standard solution (60 mL in a polypropolyne bottle).
3. One digestate 3 sample (60 mL in a polypropolyne bottle).
4. One digestate 4 sample (60 mL in a polypropolyne bottle).
5. Five standard A samples (<1 g each in 10 mL polypropolyne scintillation vials).
6. Five standard B samples (<1 g each in 10 mL polypropolyne scintillation vials).

Mean concentrations and variances [(standard deviation)^2] of the elements in the ICP standard, digestates 3 and 4, and standards A and B were calculated as the weighted means and sum of the variances of the certified standards used in the make up of each material. The calculated mean, standard deviation, and coefficient of variability (CV) values for each element in the composite standards and digestates are listed in Table 1.

3.1.2 Oily Sludge Samples

Six gas plant sludge samples were received and used in this project. and the sludges were randomly assigned a letter designation (A to F). The sludges included three sludges used in the previous phase of the study (Chow and Abboud, 1989), and three other sludges recommended by the workshop participants. Wet sludges were thawed (if frozen) and thoroughly mixed using a teflon coated mixer attached to heavy duty drill press. Subsamples were obtained (using teflon or teflon coated instruments) from

Table 1. Mean (mg/L or mg/kg), standard deviation (StDev) and coefficient of variability (CV) of the elemental contents of ICP, digestates and standard samples.

Measure	ICP	Digestate 3	Digestate 4	Standard A	Standard B
			Ag		
mean	10	2.54	2.6	142.7	153.90
StDev	1	3.45	3.5	2.9	9.20
CV(%)	10	136.00	136.0	2.0	5.97
			As		
mean	10	0.72	0.8	39.3	46.90
StDev	1	1.61	1.6	2.9	7.50
CV(%)	10	224.00	205.0	7.4	16.00
			B		
mean				142.7	153.90
StDev				2.9	20.00
C.V(%)				2.0	13.00
			Ba		
mean	10	7.63	10.7	422.0	639.00
StDev	1.	9.62	12.8	17.0	25.60
CV(%)	10	126.00	120.0	4.0	4.01
			Be		
mean	10	0.04	0.07	2.2	3.88
StDev	1	0.10	1.2	0.4	0.62
CV(%)	10	238.00	1830.0	16.1	16.00
			Cd		
mean	10	2.58	2.6	145.0	154.00
StDev	1	3.52	3.5	44.0	9.25
CV(%)	10	136.00	136.0	30.3	6.00
			Co		
mean	10	0.19	0.2	10.4	7.86
StDev	1	0.36	0.4	1.2	0.87
CV(%)	10	189.00	166.0	11.1	11.10
			Cr		
mean	10	104.00	3.6	5690.0	217.00
StDev	1	255.00	4.7	1080.0	8.70
CV(%)	10	246.00	129.0	19.0	4.00

Table 1. Mean (mg/L or mg/kg), standard deviation (StDev) and coefficient of variability (CV) of the elemental contents of ICP, digestates and standard samples.

Measure	ICP	Digestate 3	Digestate 4	Standard A	Standard B
			Cu		
mean	10	3.31	3.2	185.0	217.00
StDev	1	4.63	4.2	32.0	8.70
CV(%)	10	140.00	132.0	17.3	4.00
			Hg		
mean	10	0.29	0.3	16.2	17.30
StDev	1	0.40	0.4	12.8	1.55
CV(%)	10	138.00	135.0	79.0	8.96
			Mn		
mean	10	5.82	3.5	322.0	212.00
StDev	1	11.00	4.7	64.5	10.60
CV(%)	10	190.00	132.0	20.0	5.00
			Mo		
mean	10	2.64	2.3	148.0	163.00
StDev	1	3.58	3.7	9.2	9.80
CV(%)	10	136.00	161.0	6.2	6.00
			Ni		
mean	10	3.13	3.3	175.0	195.00
StDev	1	4.13	4.2	10.5	14.80
CV(%)	10	132.00	130.0	6.0	7.59
			Pb		
mean	10	5.21	3.0	289.0	177.00
StDev	1	6.99	3.9	20.3	8.90
CV(%)	10	134.00	132.0	7.0	5.02
			Sb		
mean	10	2.71	2.6	152.0	154.00
StDev	1	3.68	3.5	15.2	13.90
CV(%)	10	136.00	136.0	10.0	9.00
			Se		
mean	10	0.33	0.3	18.2	20.40
StDev	1	0.45	0.4	1.9	1.85
CV(%)	10	138.00	129.0	10.2	9.07

Table 1. Mean (mg/L or mg/kg), standard deviation (StDev) and coefficient of variability (CV) of the elemental contents of ICP, digestates and standard samples.

Measure	ICP	Digestate 3	Digestate 4	Standard A	Standard B
			Tl		
mean	10	2.74	2.7	144.0	180.00
StDev	1	3.02	3.0	8.7	9.00
CV(%)	10	110.00	108.0	6.0	5.00
			V		
mean	10	3.63	3.0	203.0	250.00
StDev	1	4.73	3.7	12.2	10.00
CV(%)	10	130.00	126.0	6.0	4.00
			Zn		
mean	10	9.13	3.8	504.0	225.00
StDev	1	18.30	5.0	85.7	11.30
CV(%)	10	200.00	133.0	17.0	5.02

various parts of each sludge pail during the mixing process. The dry sludge samples were subsampled after several mixing and homogenization operations using a process similar to soil mixing preparations.

Sludges were transferred to either polypropolyne scintillation vials (<1 g), or to 60 mL polypropolyne bottles, capped, packaged with the standard samples and shipped to the participating laboratories. Each of the participating laboratories was shipped the following sludge samples:

1. Six sludge A samples (<1 g each in 10 mL polypropolyne scintillation vials).
2. Six sludge B samples (<1 g each in 10 mL polypropolyne scintillation vials).
3. Six sludge C samples (<1 g each in 10 mL polypropolyne scintillation vials).
4. Six sludge D samples (<1 g each in 10 mL polypropolyne scintillation vials).
5. Six sludge E samples (<1 g each in 10 mL polypropolyne scintillation vials).
6. Six sludge F samples (<1 g each in 10 mL polypropolyne scintillation vials).
7. One sludge A sample (60 mL in a polypropolyne bottle).
8. One sludge B sample (60 mL in a polypropolyne bottle).
9. One sludge C sample (60 mL in a polypropolyne bottle).
10. One sludge D sample (60 mL in a polypropolyne bottle).
11. One sludge E sample (60 mL in a polypropolyne bottle).
12. One sludge F sample (60 mL in a polypropolyne bottle).

3.2 METHODS

The reported methods used for the analysis varied for the various participating laboratories. The workshop had recommended using the CPA-ARC method plus another that is commonly used by each of the participating laboratories. Furthermore, it was recommended that all elements (19) be analyzed for in all the samples supplied.

After the results were returned to our laboratory, it became apparent that the workshop recommendations were not followed in their entirety. Adherence to the recommendations varied widely from some labs that did not attempt the CPA-ARC method to others that did it with some modifications. Some labs did only selected elements and still others did selected samples. Documentation about the methods (other than the CPA-ARC method) used and modifications of the CPA-ARC method were not requested and were not supplied by the laboratories. In our data analysis we grouped the methods that were similar to the CPA-ARC method as such and the rest as others.

3.3 STATISTICAL AND GRAPHICAL ANALYSIS

Statistical analysis of the accumulated data was done by constructing and comparing control charts of the various standard and sludge samples. More in-depth analysis of the data was precluded by the small number of the participating laboratories and the uneven distribution of the results reported for the various elements.

Data management and graphics were done using the database management software RS1 (BBN, 1987) running on a VAX 780 computer. Statistical analysis of the data (summary statistics) were also done with the RS1 statistical package on the VAX computer.

4. RESULTS AND DISCUSSIONS

The results of this round robin will be presented and discussed for each element and for cyanide individually. The various laboratories will be assessed relative to the recovery (within confidence limits) of elements for standard samples and good duplication for all samples.

4.1 RECOVERIES OF TRACE ELEMENTS AND CYANIDE FROM STANDARDS AND SLUDGES

The recovery of elements from the ICP standard, digests, standards, and sludges will be treated graphically as quality control charts and are presented in Figures 1 to 204. These graphs show the distribution of the analytical results (for all the participating laboratories and the method used) around the expected mean value of the analyte and bounded by the upper and lower control and warning limits. The plots are more commonly referred to as Shewhart or quality control charts (Dux, 1986). The control charts (Figures 1 to 204) show the 99% confidence interval of the standard data as the area between the upper (UCL, mean + 3 s) and lower (LCL, mean - 3 s) control limits, and the 95% confidence interval as the area between the upper (UWL, mean + 2 s) and the lower (LWL, mean - 2 s) warning limits (Dux, 1986).

The means and standard deviations of the ICP standard, digest and standards are reported in the materials and methods section of this report. The means and standard deviation of the six sludges were calculated for each element and cyanide and are reported with several other statistical variables in tables 2 to 21.

The data received from all the laboratories are presented in Appendix 1. This data includes all elemental and cyanide concentrations in all the tested materials.

The following sections will examine in detail the data for each element and cyanide and discuss the results of the different methods and laboratory combinations. These discussions will include the graphical and statistical data presented, and comments on the similarities and differences between the laboratories and the methods used.

4.1.1 TRACE ELEMENTS

4.1.1.1 Ag

Four labs reported Ag for the digestate and standard samples. The results for Digestate 3 (Figure 1) show that all labs, except for lab 5 were within the 95% confidence interval. Digestate 4 data (Figure 2) indicate that all the labs were within the 95% confidence interval. These results indicate good agreement in the analysis of digestate solutions between most of the labs.

The data for standards A and B are reported in Figures 3 and 4 respectively. Figure 3 shows that all the labs were outside the 99% confidence interval irrespective of the methods used. Figure 4 shows that 3 out of the 4 labs had some data within the 99% confidence interval, and the data from the only lab outside of the control limits varied by an order of magnitude. The results for these standards show poor recovery of Ag for most labs irrespective of the method used. The slightly better results for standard B could be due to its wider control limits.

The statistical data for Sludges A to F are shown in Table 2, and their control charts in Figures 5 to 10. Table 2 shows that the results (above detection) reported were for 3 sludges only (A, B and D). The coefficient of variation of the sludges varied from 15 to 84% and it seems to be related to the number of reported results.

Figure 5 indicates that 2 above and 6 below detection values were reported for sludge A. The below detection values varied by more than an order of magnitude. Figure 6 indicates that 3 labs reported below detection values for sludge B, while one lab reported 6 values clustered together. The low CV for this sludge is indicative of the data usually reported from a single lab. Figure 7 indicates that all the labs reported below detection values for sludge C. Figure 8 shows that 2 labs reported above detection limits for sludge D, with the results varying from 3 to 13 mg/kg. Figure 9 indicates that all the labs reported below detection values for sludge E. Figure 10 shows that only one above detection value was reported for sludge F, with several below detection values varying by more than an order of magnitude.

Figure 1. Mean and Confidence Intervals for Ag in Digestate 3

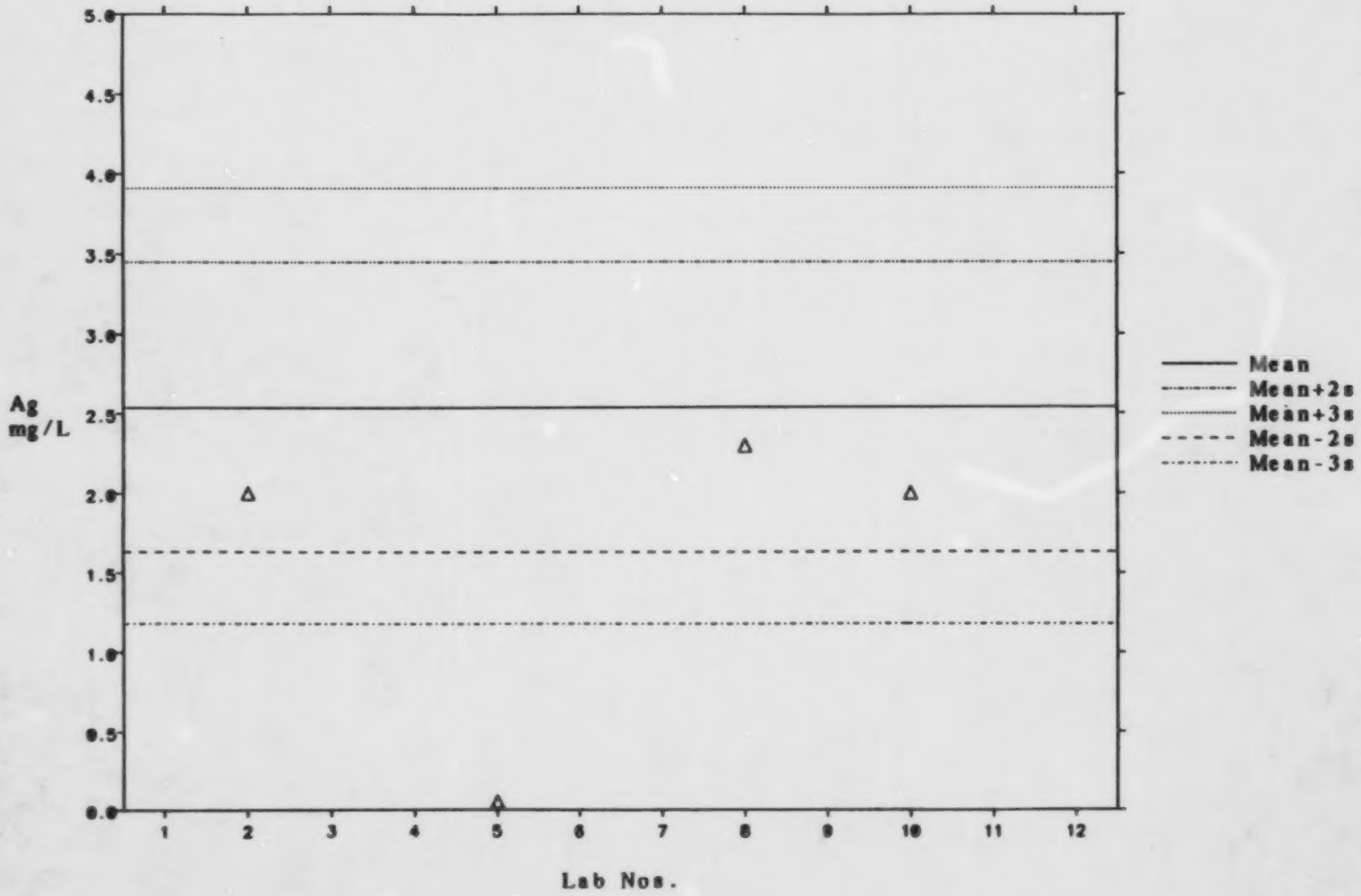


Figure 2. Mean and Confidence Intervals for Ag in Digestate 4

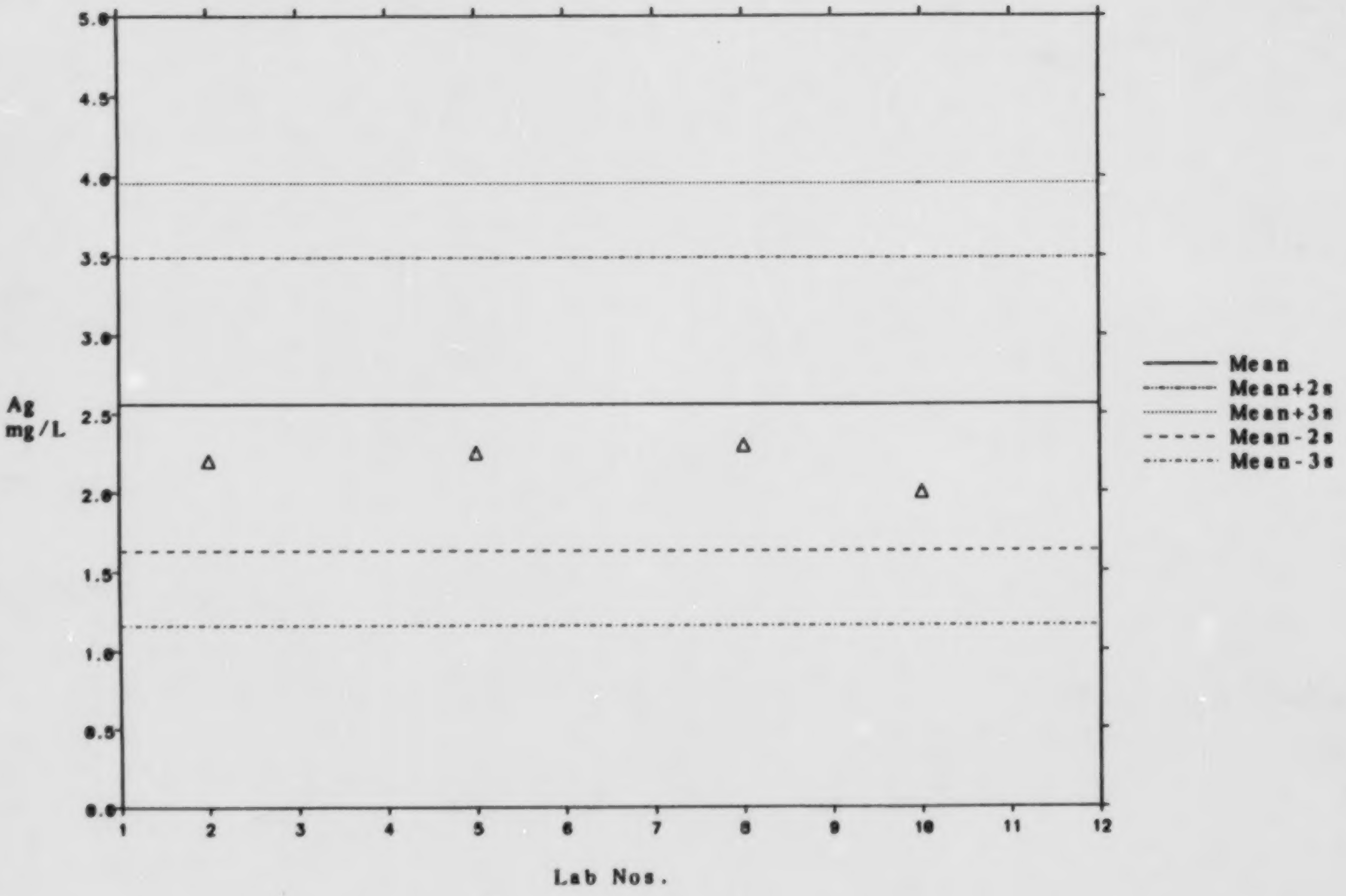


Figure 3. Mean and Confidence Intervals for Ag in Standard A

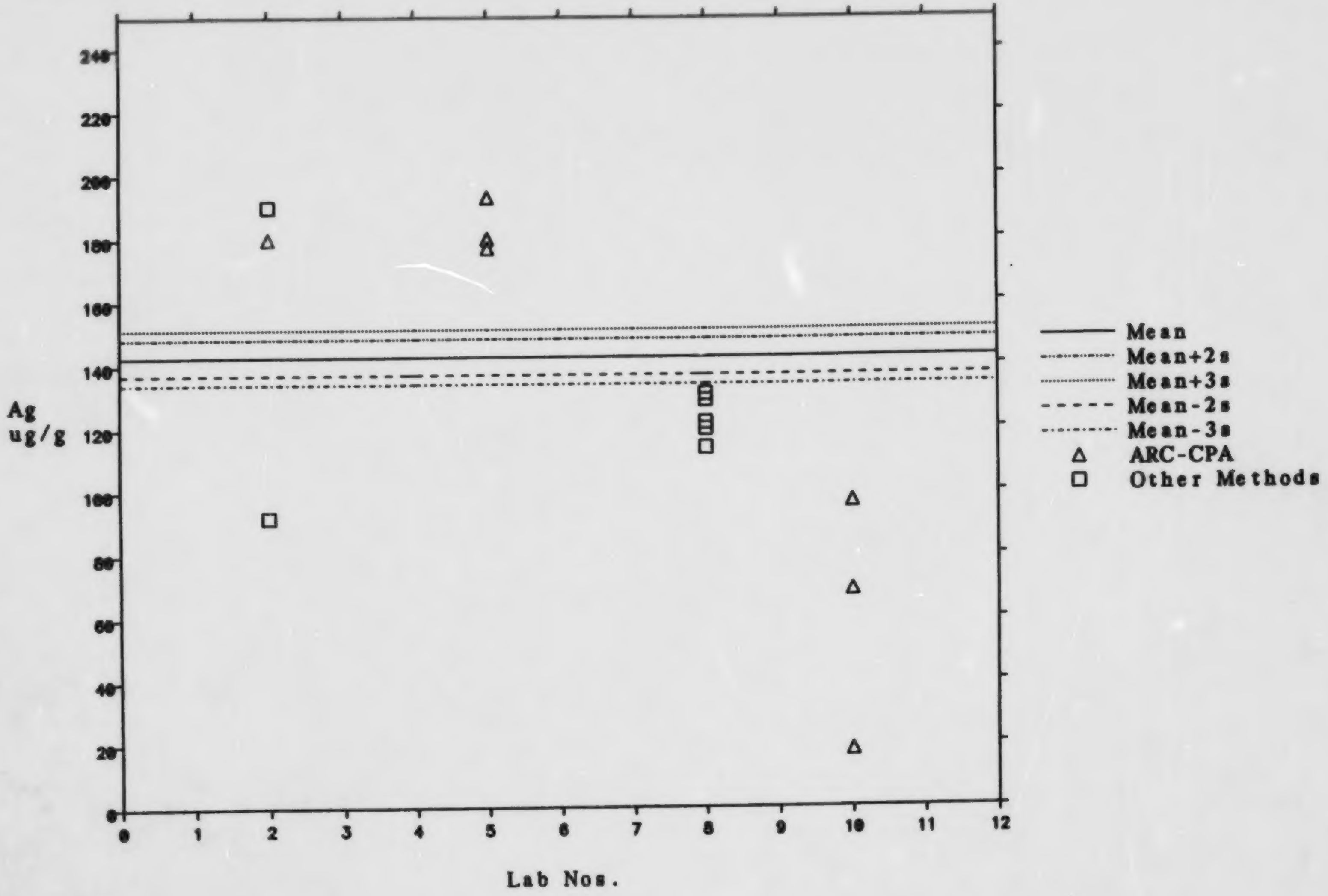


Figure 4. Mean and Confidence Intervals for Ag in Standard B

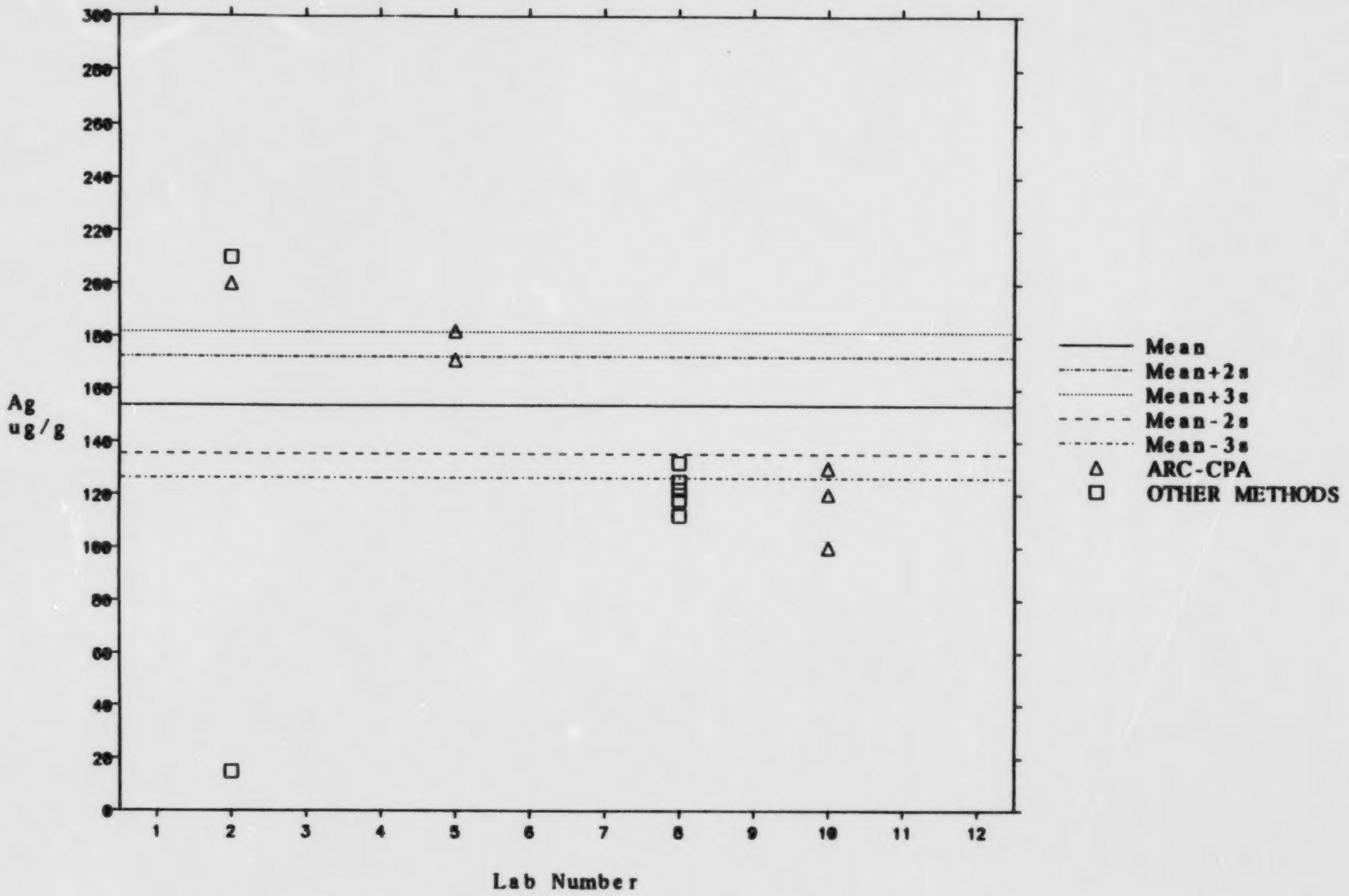


Table 2. Statistical Parameters for Ag (mg/Kg) in Sludges A-F.

Statistic	A	B	C	D	E	F
Count	2.00	6.000	0	4.000	0	1
Sum	6.50	14.400		29.900		5
Mean	3.25	2.400		7.480		5
SEM	0.75	0.144		3.080		
Median	3.25	2.400		5.500		5
Variance	1.13	0.124		38.000		
StDev	1.06	0.352		6.160		
Maximum	4.00	3.000		16.000		5
Minimum	2.50	2.000		2.900		5
Range	1.50	1.000		13.100		0
Skewness		0.866		1.240		
Kurtosis		1.190		0.653		
C.V(%)	32.60	14.700		82.400		

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 5. Analysis of Ag in Sludge A Sample

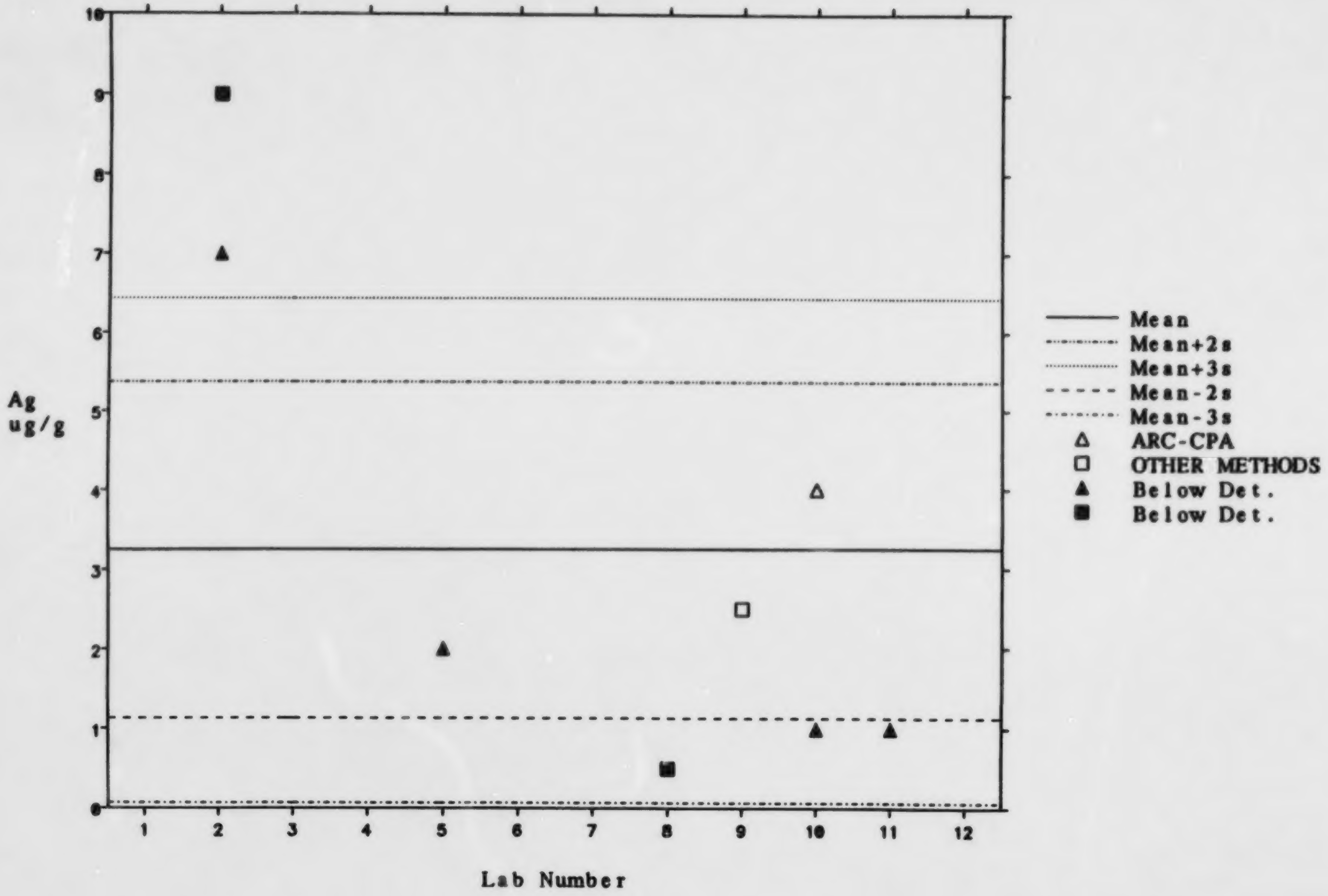


Figure 6. Analysis of Ag in Waste B

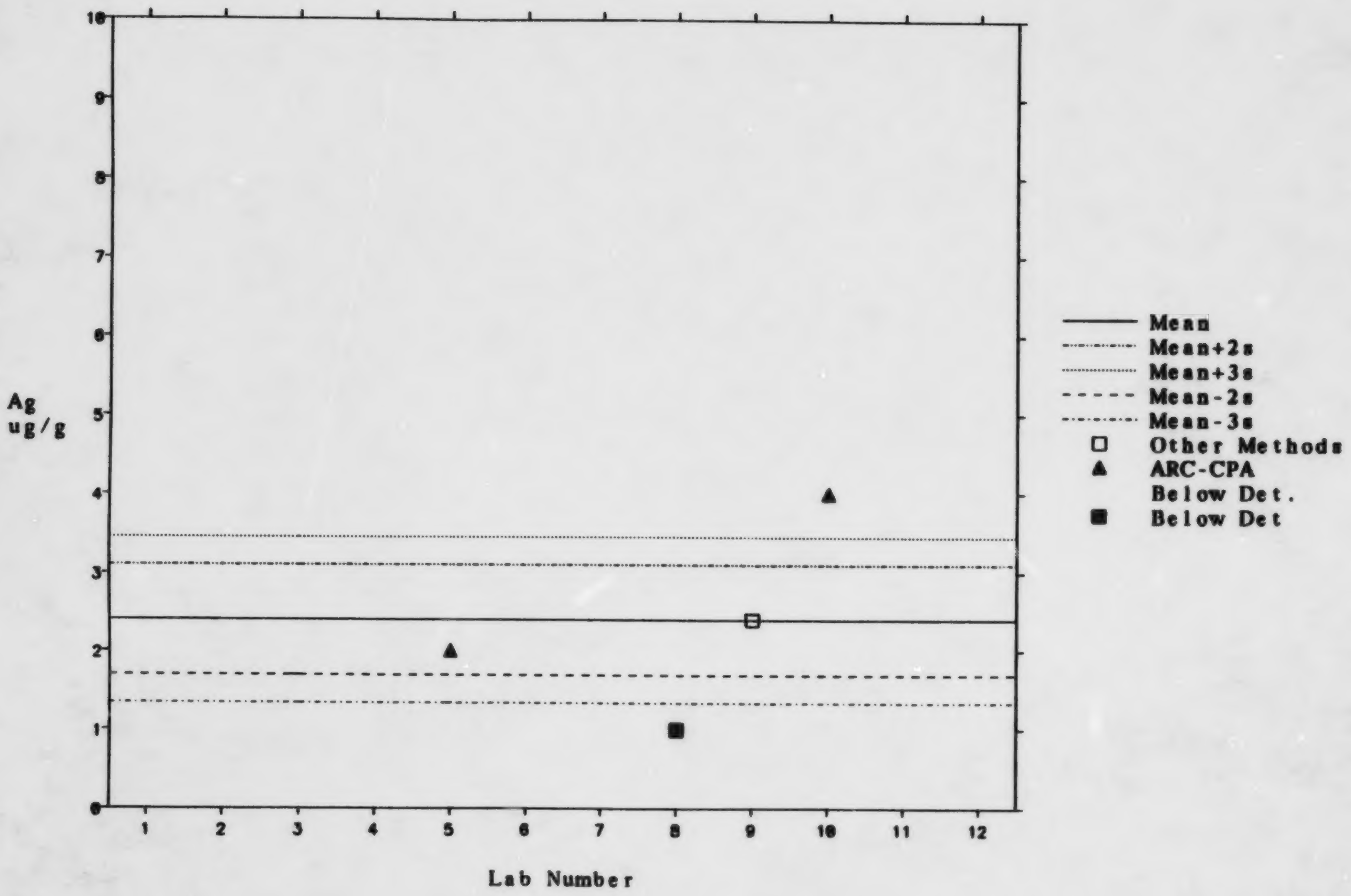


Figure 7. Analysis of Ag in Sludge C

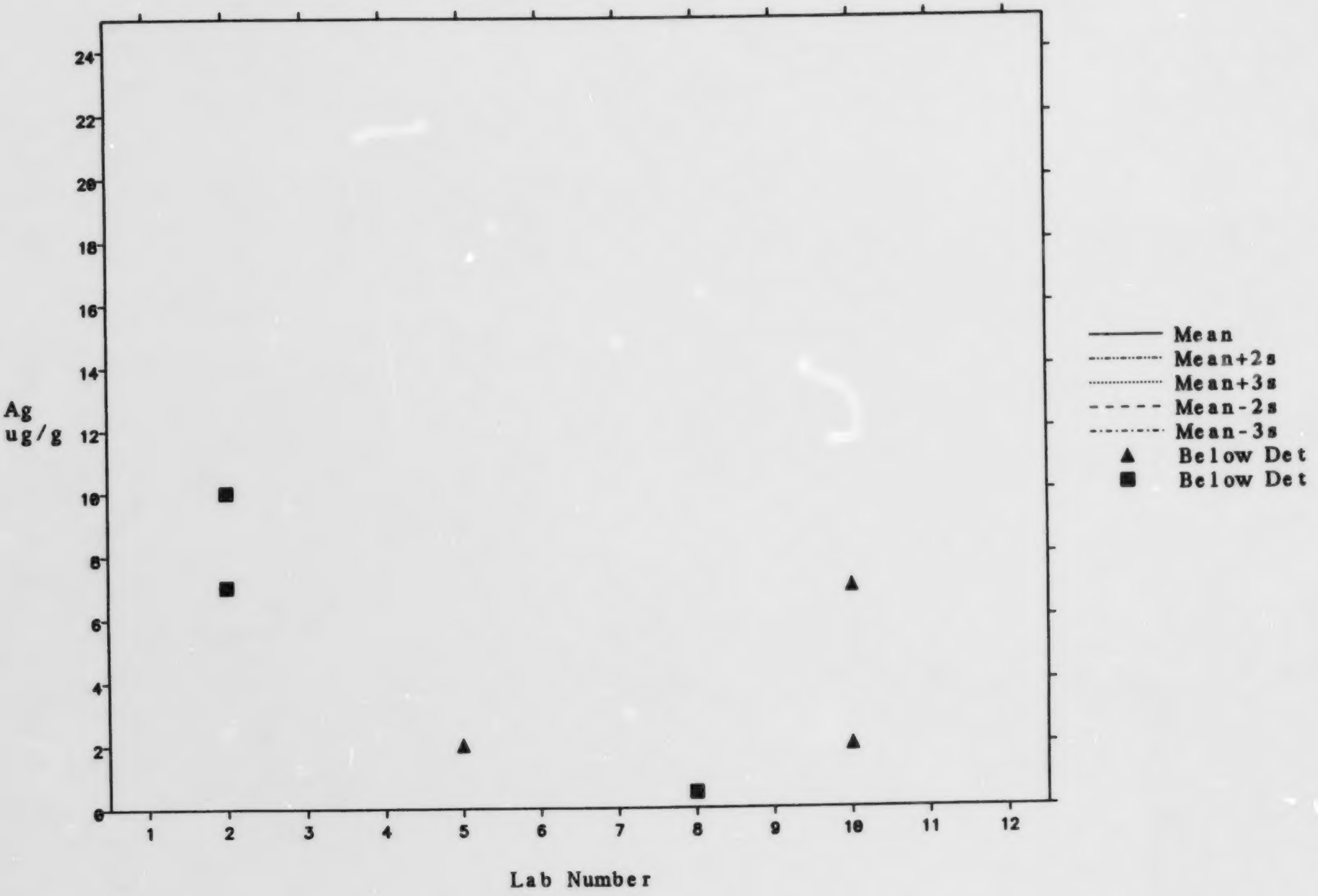


Figure 8. Analysis of Ag in Sludge D

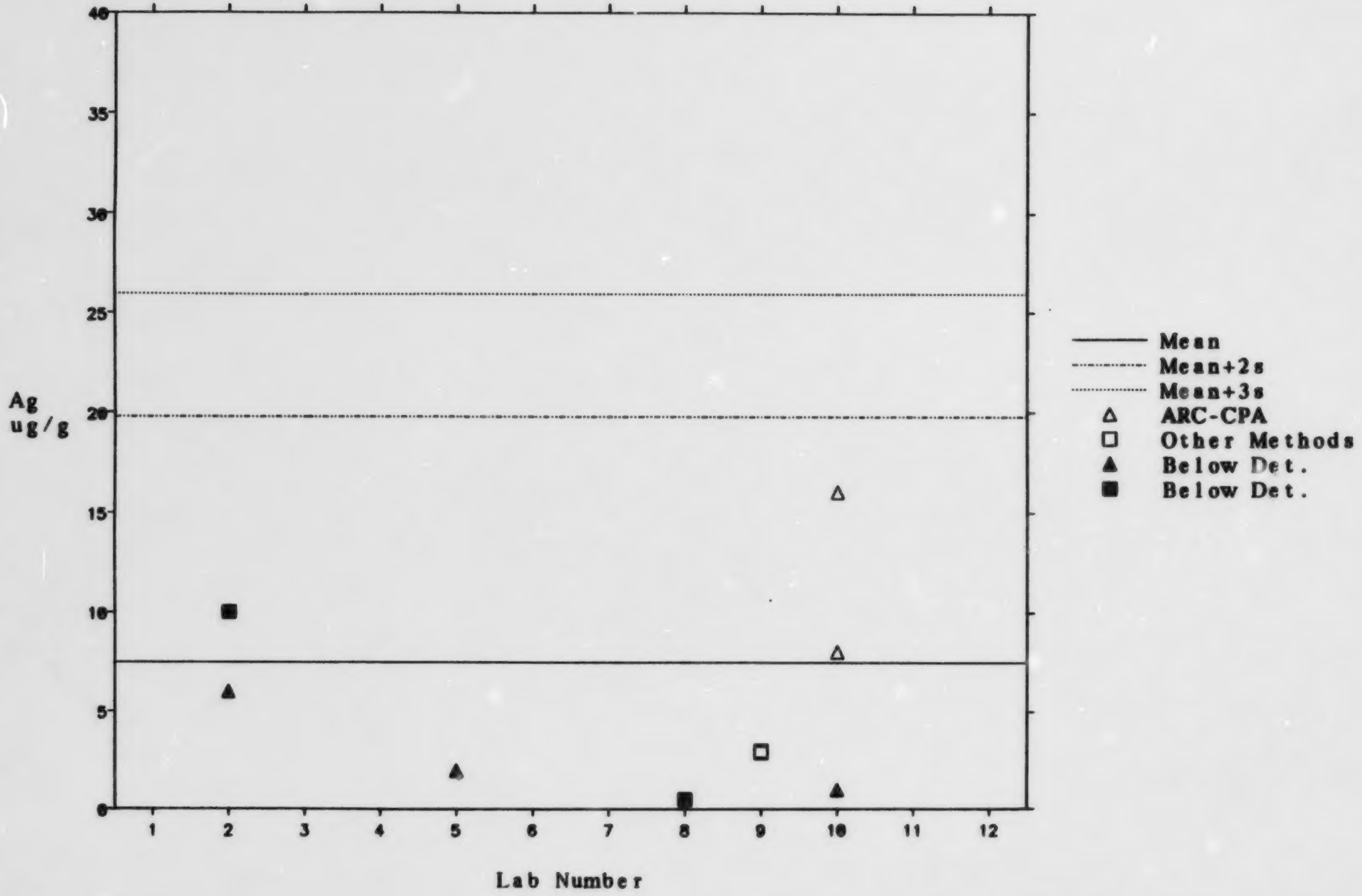


Figure 9. Analysis of Ag in Sludge E

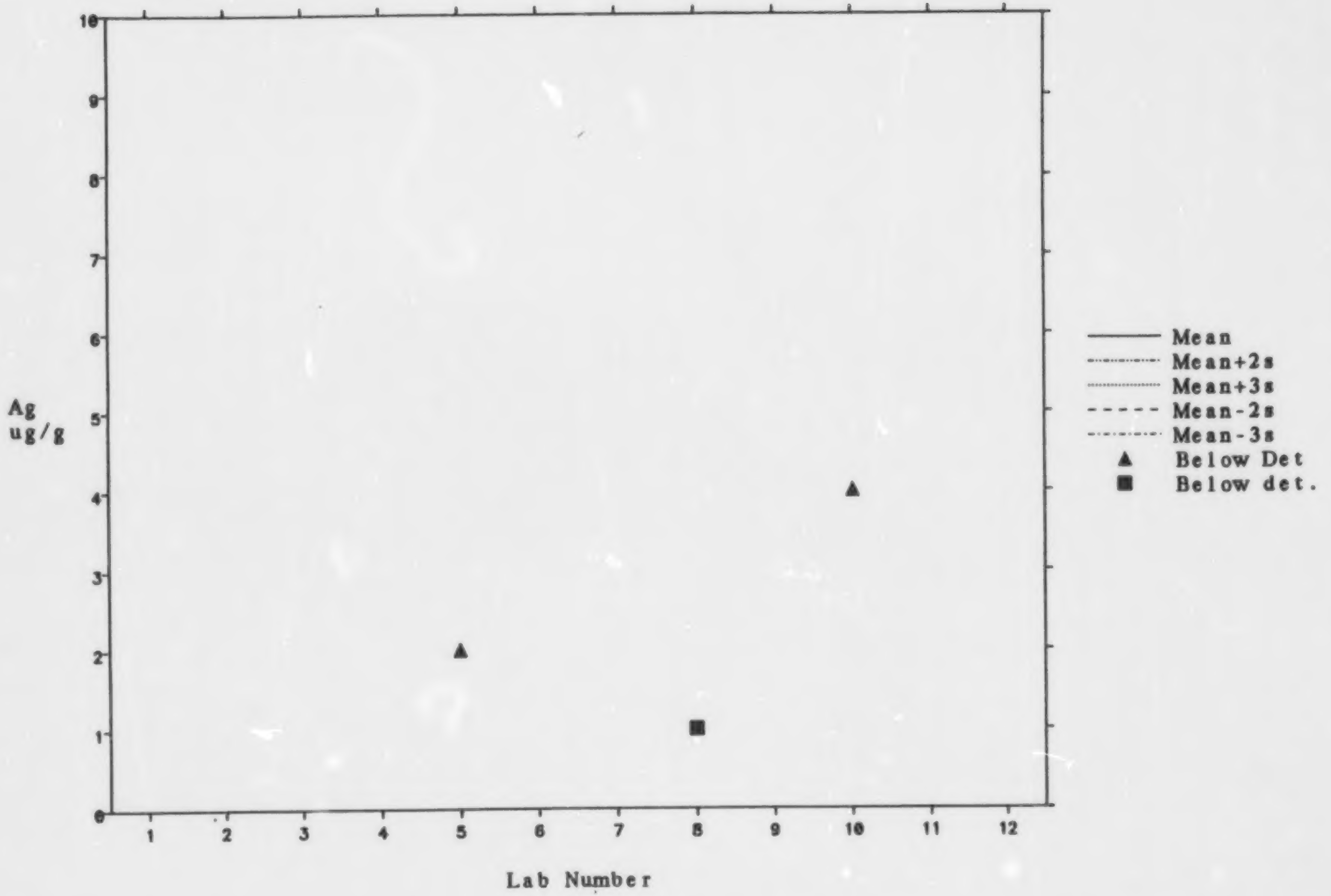
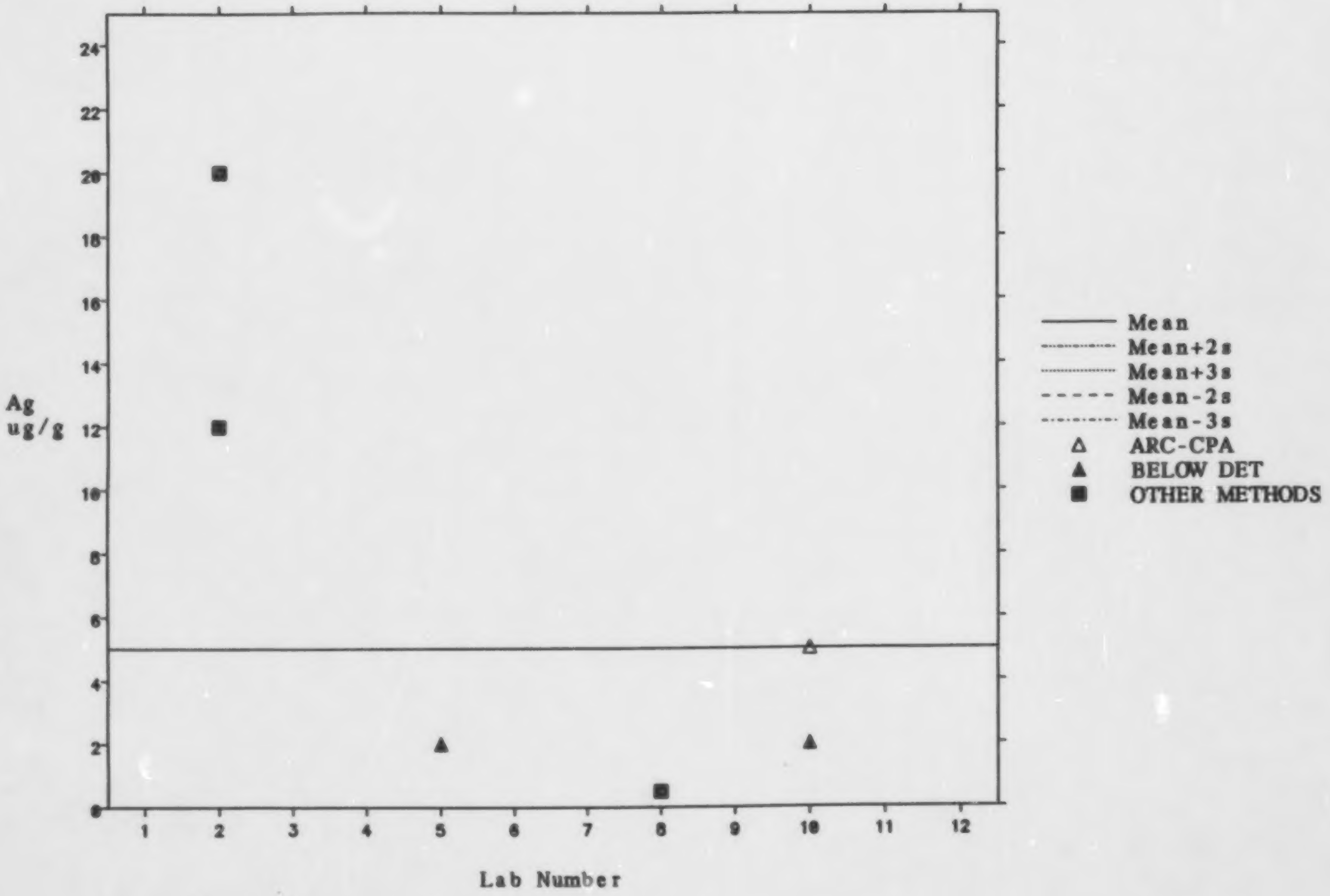


Figure 10. Analysis of Ag in Sludge F



These results show the very wide variation in the detection limits for Ag reported by the various labs. This, no doubt contributed to the observed scatter in the reported data. No further conclusions could be made from these analyses because of the scarceness of the reported data.

4.1.1.2 As

Three labs reported As in the ICP standard (Figure 11), two of which were very near the mean value and the other much higher than the control limit. Four labs reported As for the digestate samples (Figures 12 and 13), with acceptable results bound by the upper and lower warning limits. Five labs reported As for the standard A and B samples (Figures 14 and 15). Standards A and B values (Figures 14 and 15) show that all labs had data within the control limits, with 3 labs reporting some data both inside and outside the control limits. Both digestion methods showed data inside the acceptable limits, with the outlier data from the ARC-CPA method only.

The statistical data for sludges A to F are shown in Table 3, and their control charts in Figures 16 to 21. Table 3 shows that results (above detection) were reported for all sludges, with number of analyses varying from 7 to 23 samples. The mean As values ranged from 3.3 mg/kg for sludge E to 16.1 for sludge B. The coefficient of variation of the sludges varied from 55 to 180% and it seems to be inversely related to the number of reported results.

Figures 16 to 21 indicate that all the reported As data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods. The detection limits for As varied widely between labs, with differences higher than one order of magnitude.

The As data shows better agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed, with lower CV attained when more samples are analyzed.

Figure 11. Mean and Confidence Intervals for As in an ICP Standard

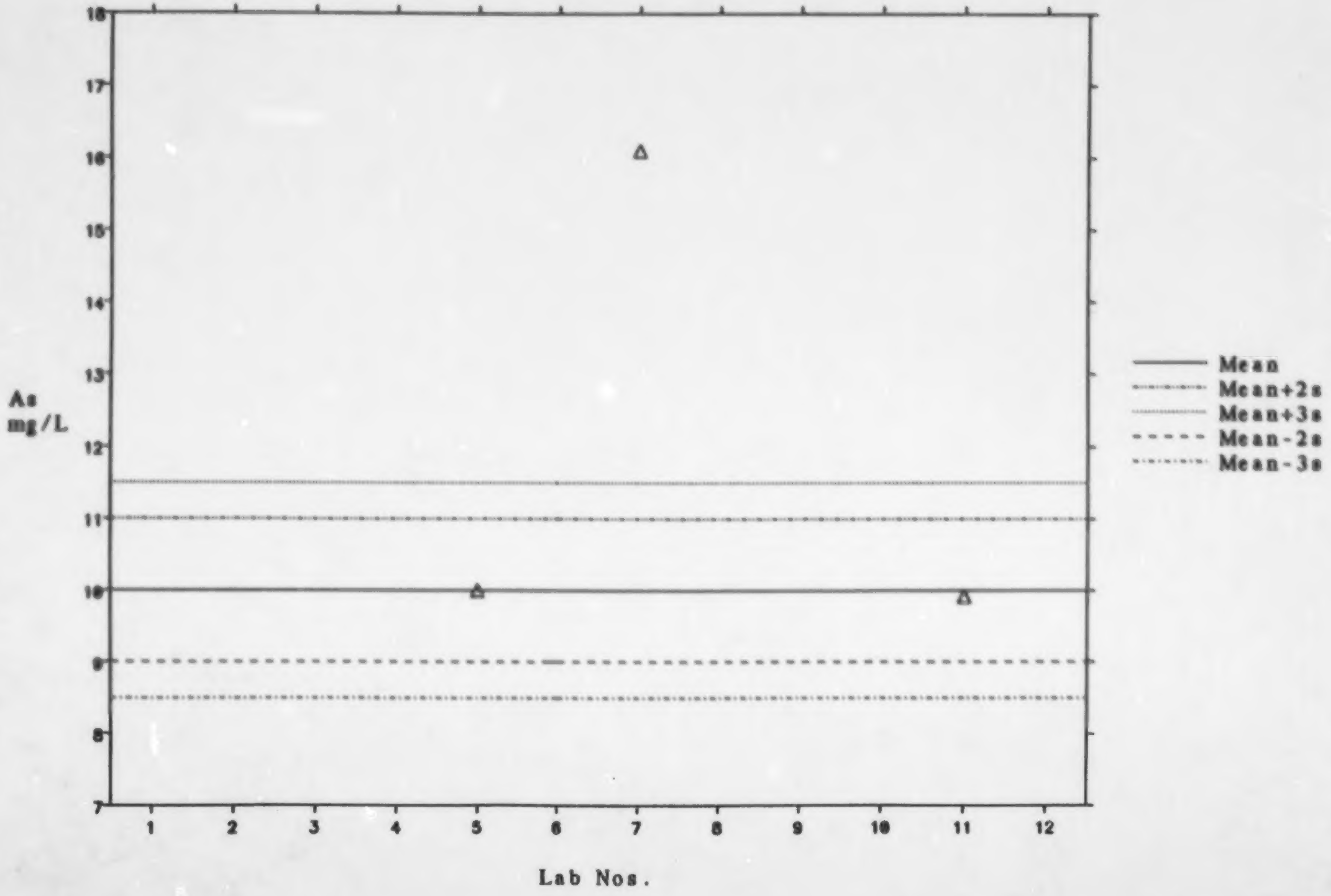


Figure 12. Mean and Confidence Intervals for As in Digestate 3

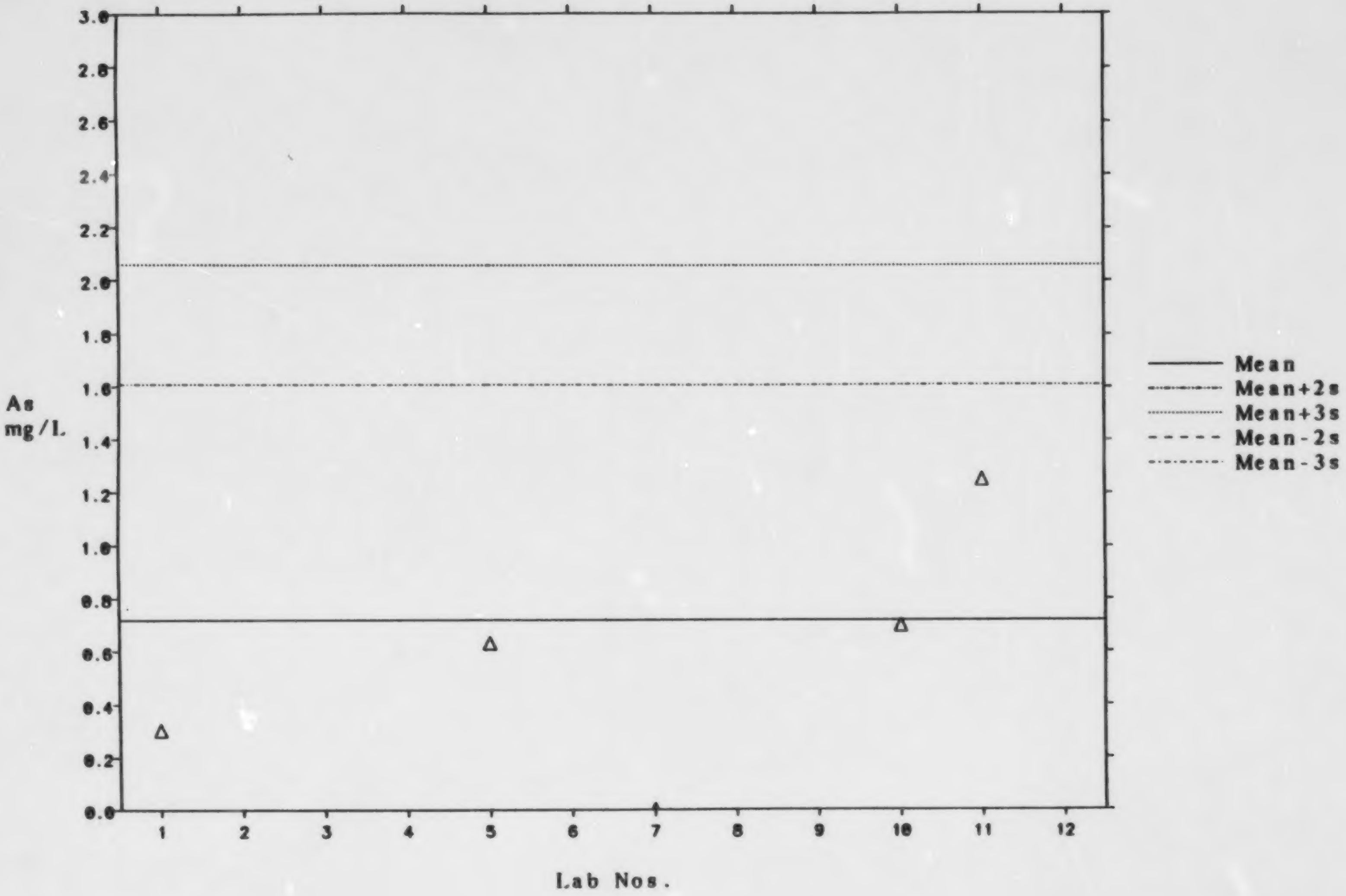


Figure 13. Mean and Confidence Intervals for As in Digestate 4

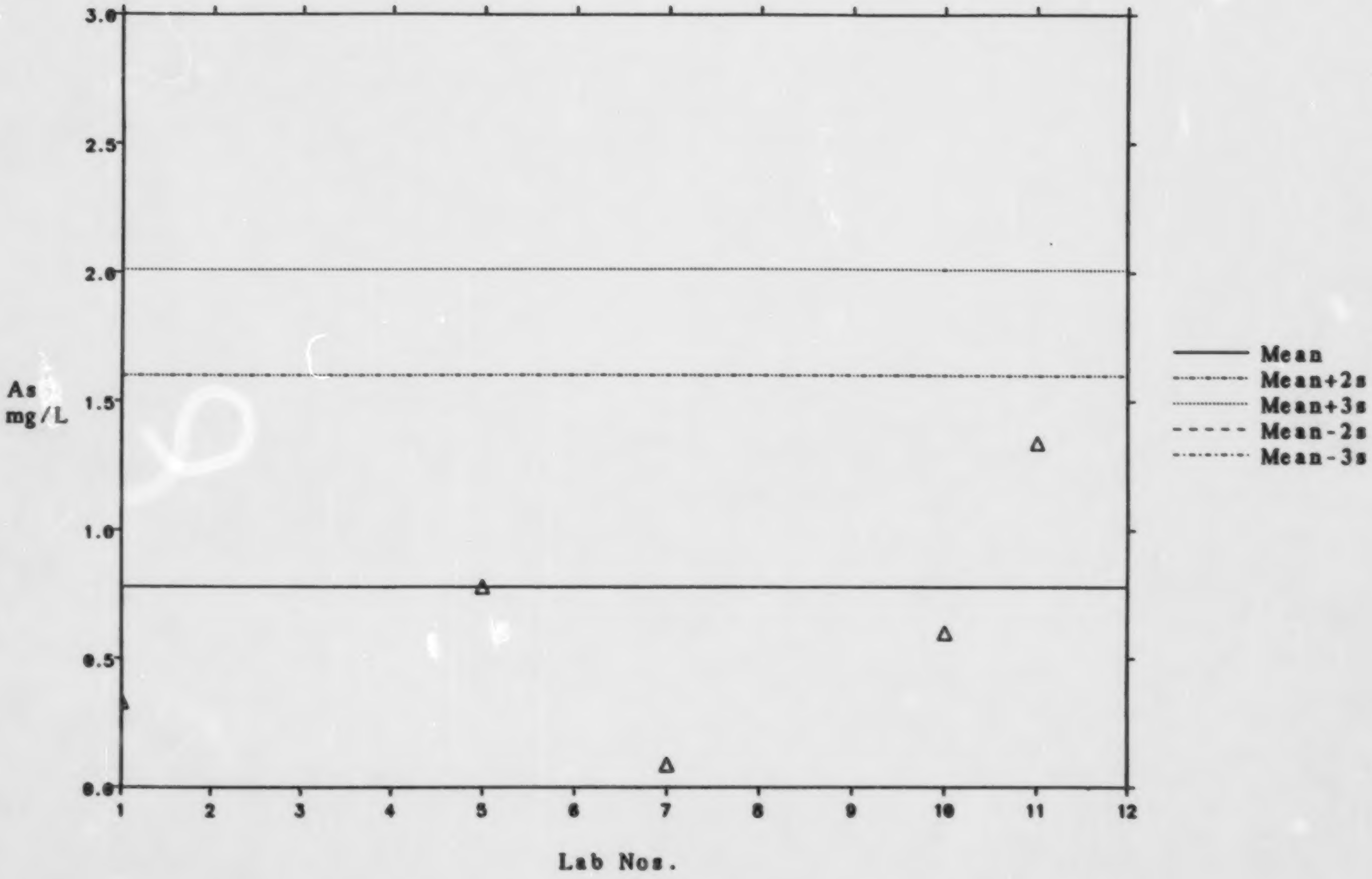


Figure 14. Mean and Confidence Intervals for As in Standard A

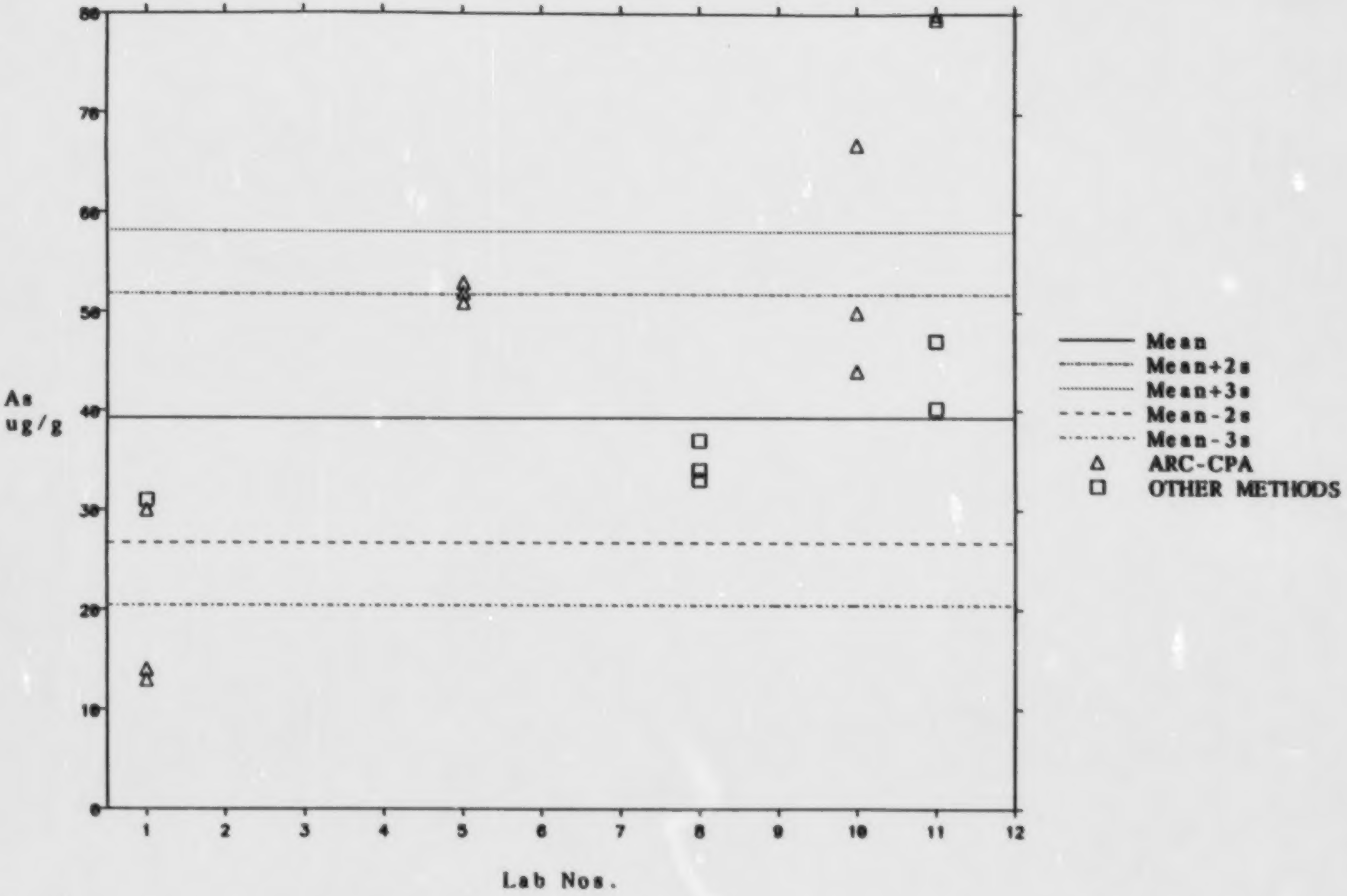


Figure 15. Mean and Confidence Intervals for As in Standard B

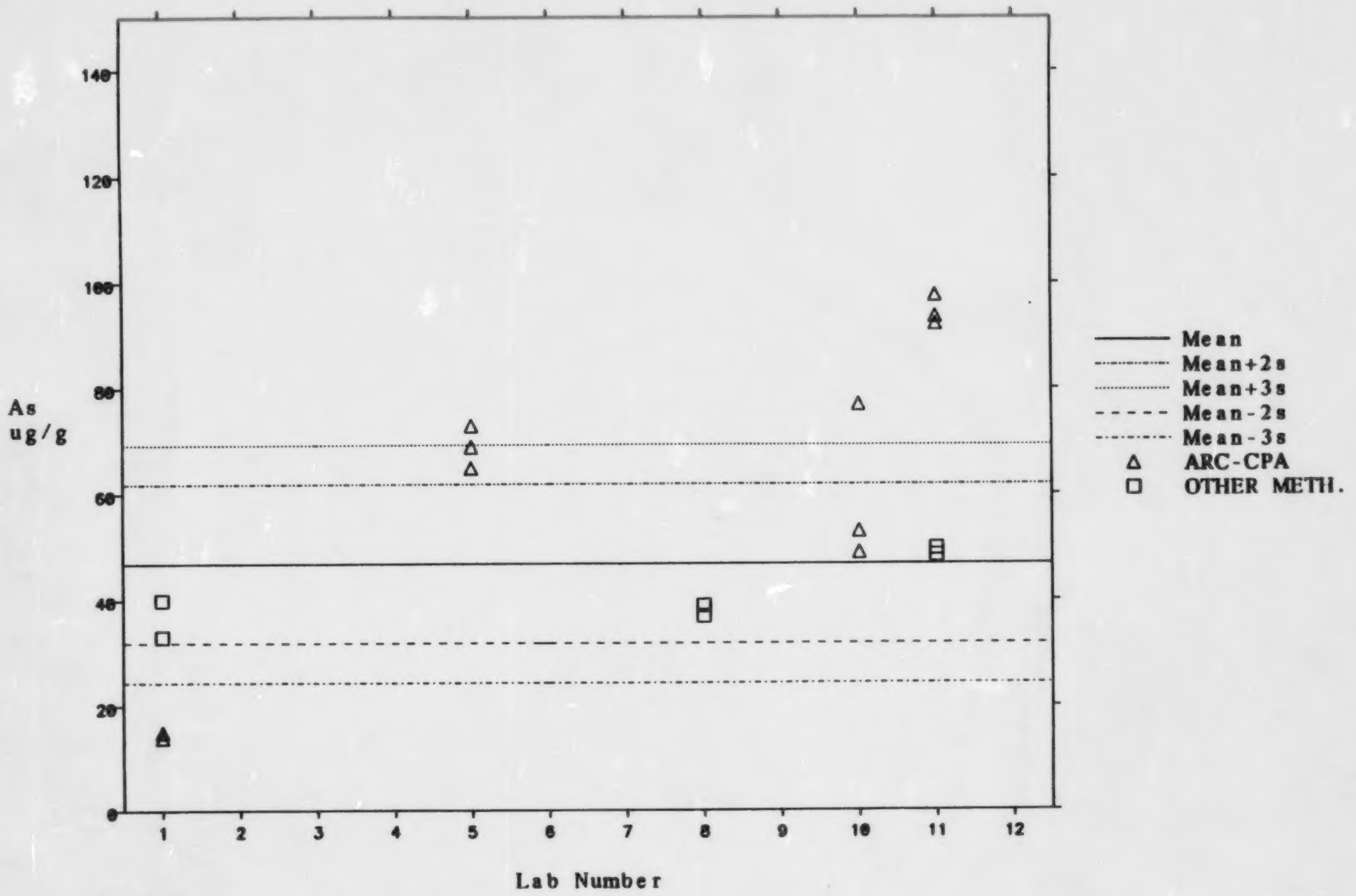


Table 3. Statistical Parameters for As (mg/Kg) in Sludges A-F.

Statistic	A	B	C	D	E	F
Count	23.00	20.00	27.00	21.00	7.00	19.00
Sum	115.00	322.00	184.00	214.00	23.40	205.00
Mean	5.01	16.10	6.81	10.20	3.34	10.80
SEM	0.57	2.66	1.06	1.10	2.28	1.76
Median	5.00	9.95	3.90	10.10	0.07	9.80
Variance	7.53	141.00	30.40	25.20	36.20	59.10
StDev	2.74	11.90	5.51	5.02	6.02	7.69
Maximum	13.00	38.20	18.00	20.00	16.40	28.00
Minimum	1.60	4.10	0.60	2.30	0.03	1.70
Range	11.40	34.10	17.40	17.70	16.40	26.30
Skewness	0.93	0.83	0.84	0.25	2.23	0.77
Kurtosis	1.75	-0.78	-0.92	-0.39	5.09	-0.29
C.V(%)	54.80	73.90	81.00	49.30	180.00	71.30

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 16. Analysis of As in Sludge A

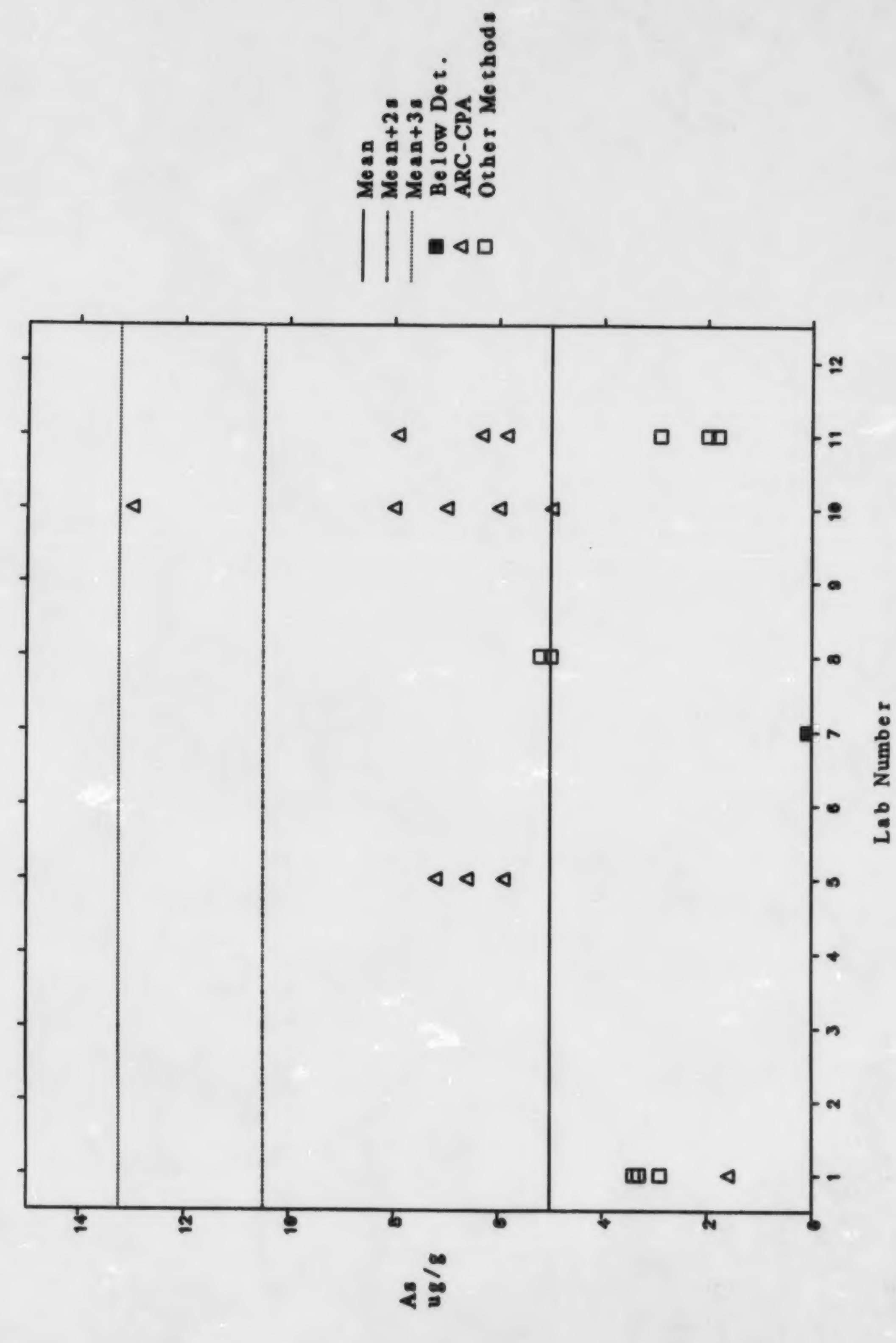


Figure 17. Analysis of As in Sludge B

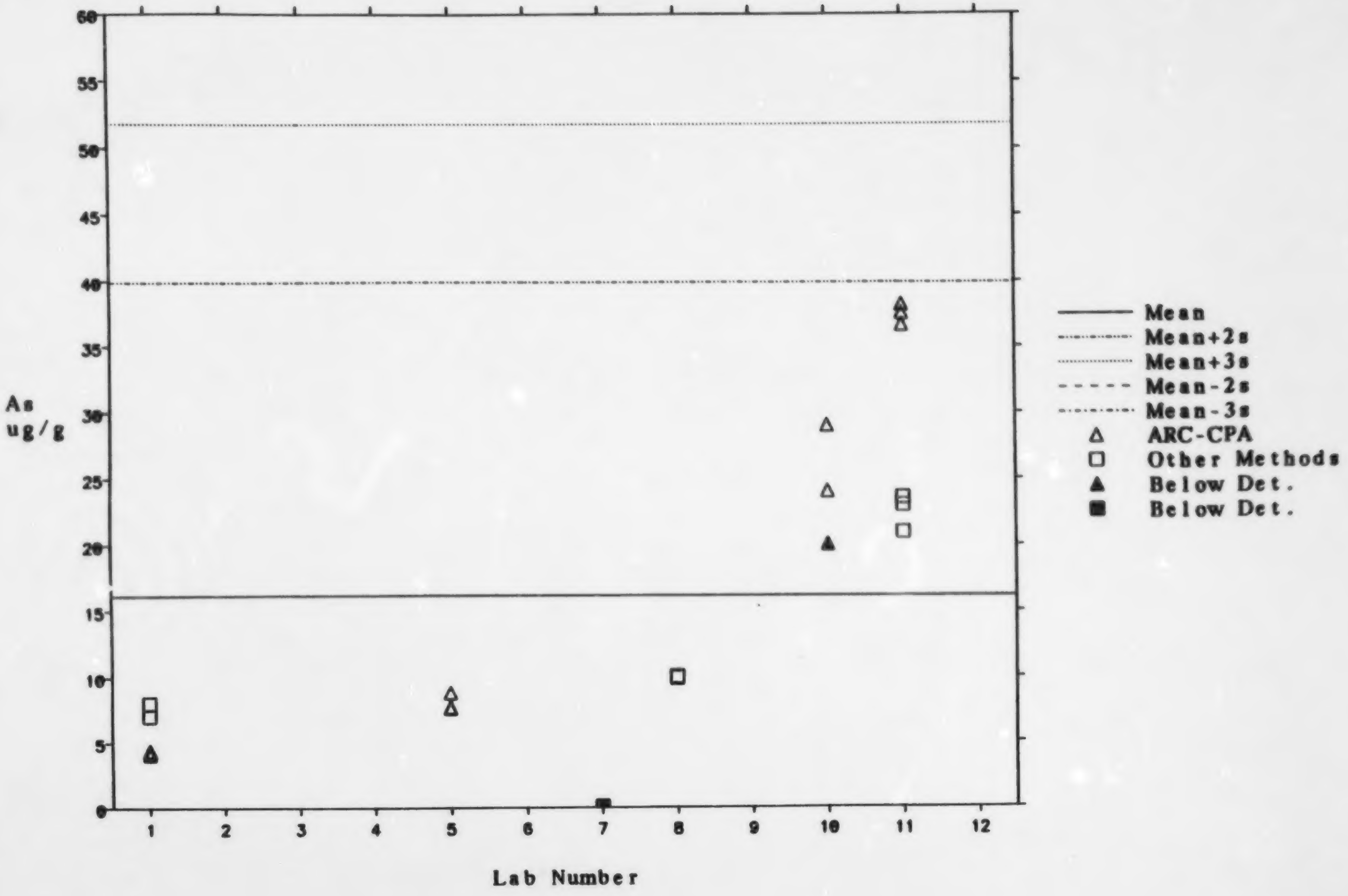


Figure 18. Analysis of As in Sludge C

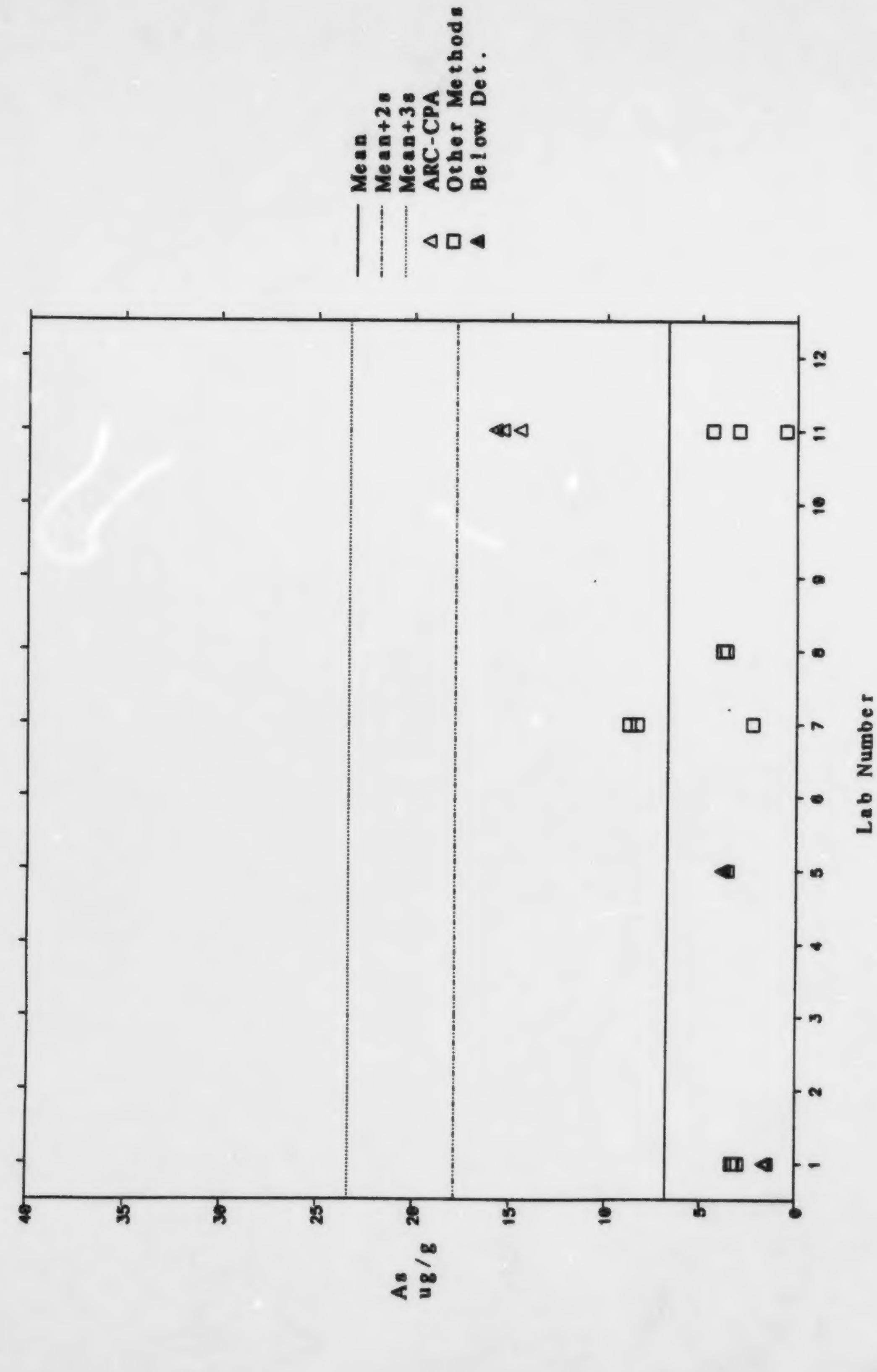


Figure 19. Analysis of As in Sludge D

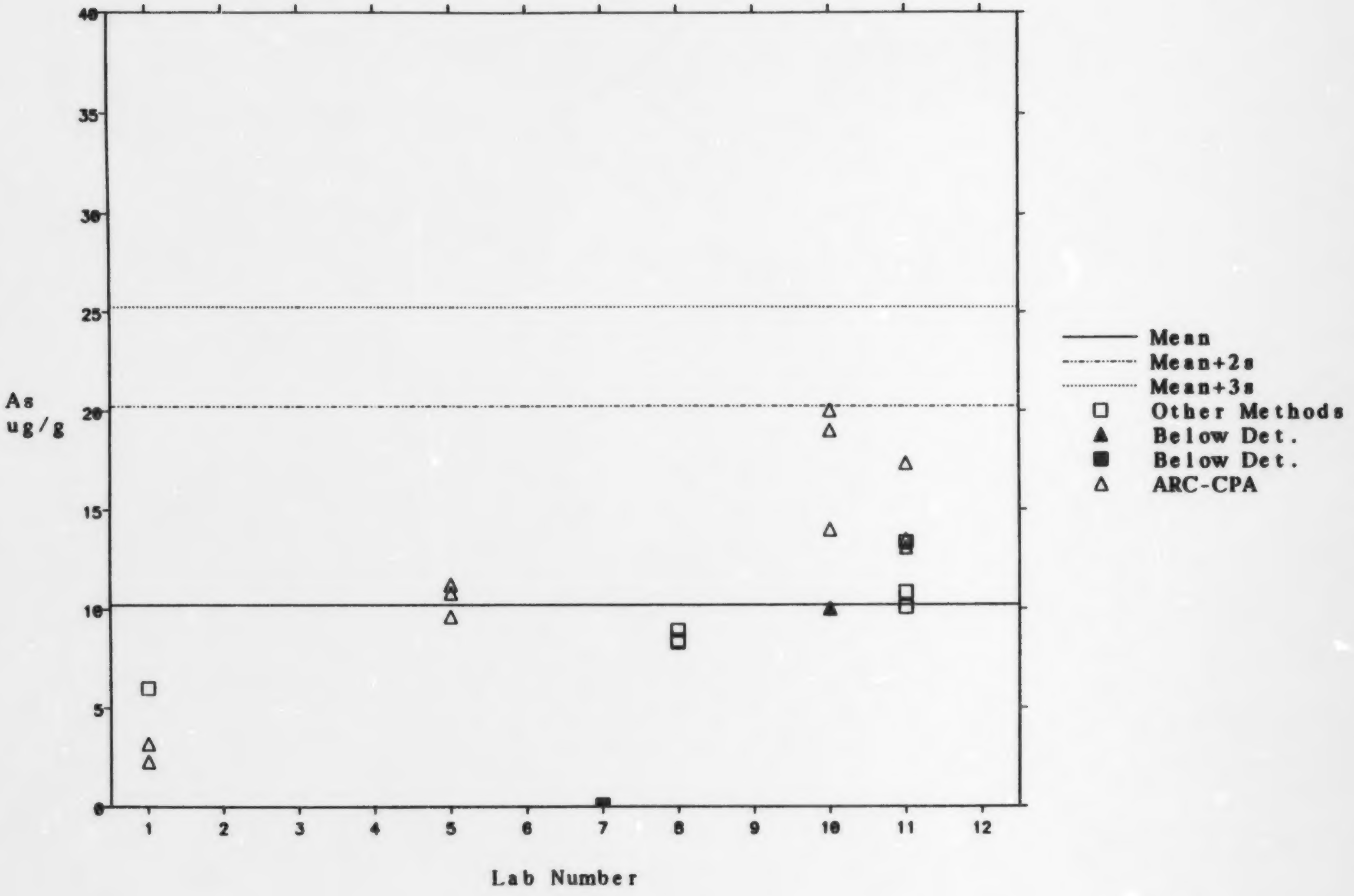


Figure 20. Analysis of As in Sludge E

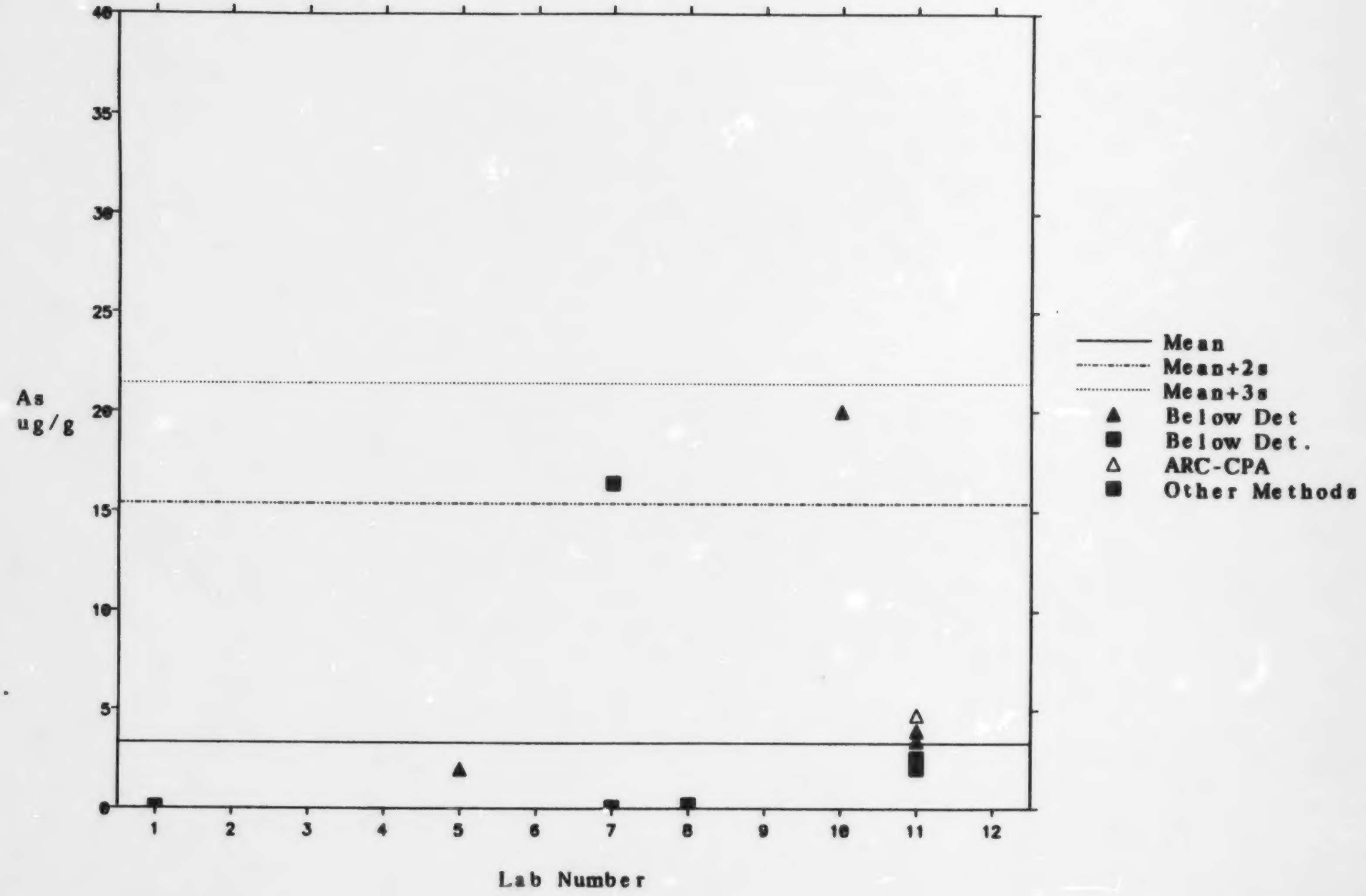
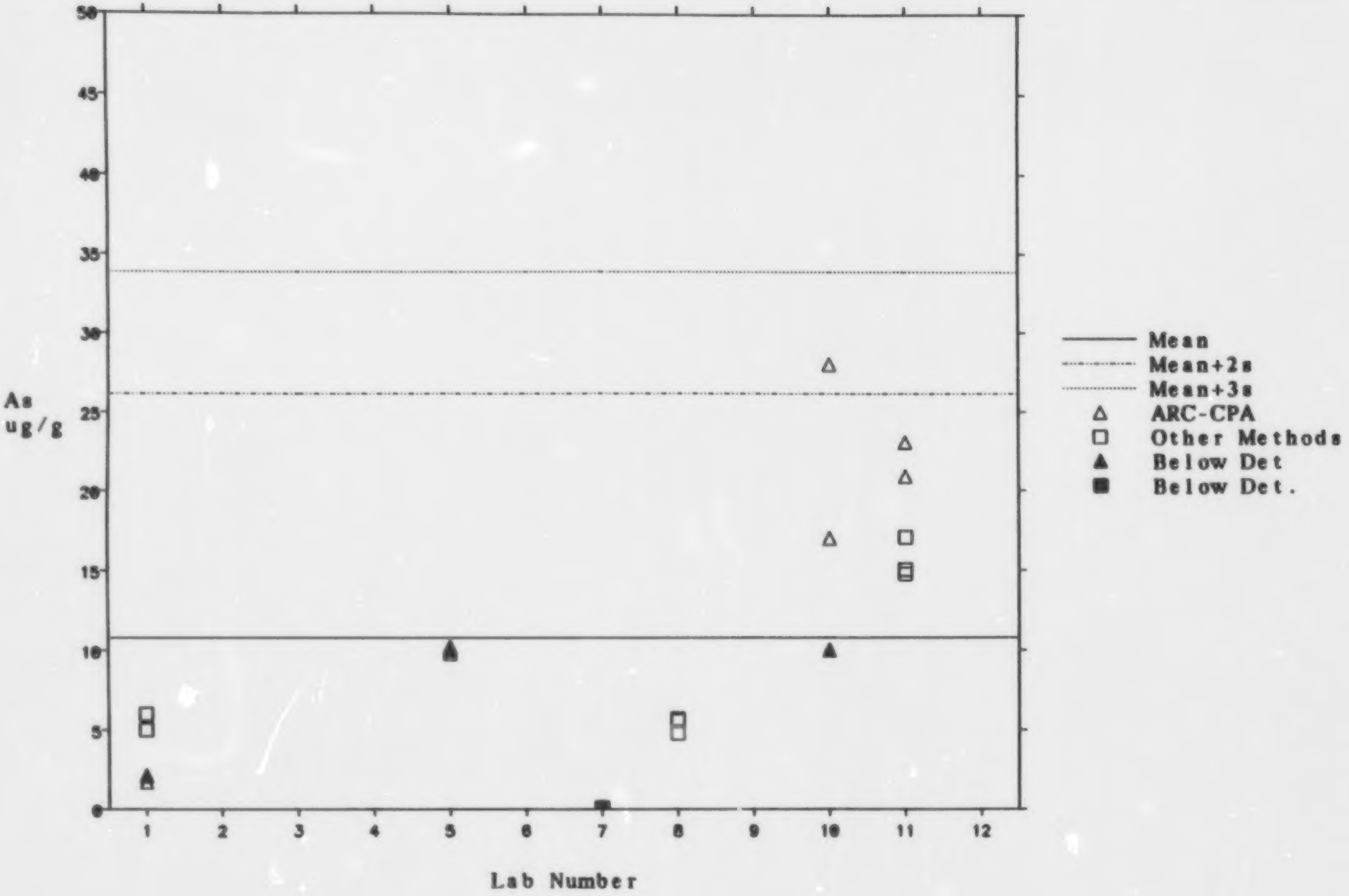


Figure 21. Analysis of As in Sludge F



4.1.1.3 B

The statistical data for B in sludges A to F are shown in Table 4, and their control charts in Figures 22 to 27. Table 3 shows that results (above detection) were reported for all sludges, but only two labs participated with the total number of analyses varying from 4 to 6 samples. The mean B values ranged from 3.9 mg/kg for sludge E to 87.4 for sludge B. The coefficient of variation of the sludges varied from 67 to 112% and it seems to be partly due to the small number of reported results.

Figures 22 to 27 indicate that all the reported B data fell within the sludges' control limits, but this has to be assessed with much cation as results were available from only two labs.

The very small number of samples and labs in the B data precludes any significant conclusion.

4.1.1.4 Ba

Seven labs reported Ba in the ICP standard (Figure 28), all but two labs were within the control limits. One of the two outlier labs was between the control and warning limit, while the other was significantly lower than the lowest warning limit. Eight labs reported Ba for the digestate samples (Figures 29 and 30). Digestate 3 data (Figure 29) were all within the control limits, while two labs were outside the warning limits of Digestate 4 (Figure 30). Six labs reported Ba for the standard A and five labs for the standard B samples (Figures 31 and 32). Figure 31 shows that 3 labs reported data within Standards A's warning limit and the other 3 were outside those limits. Standard B values (Figure 32) show that all but one lab had data within the warning limits, with 2 labs reporting some data both inside and outside the control limits. Figure 32 also shows that the results of the ARC-CPA method were more frequently located within the warning limits than the results of other methods.

Table 4. Statistical Parameters for B (mg/Kg) in Sludges A-F.

Statistic	A	B	C	D	E	F
Count	6.00	5.0	4.000	5.00	4.000	6.0
Sum	124.00	437.0	42.000	116.00	15.700	183.0
Mean	20.70	87.4	10.500	23.20	3.930	30.5
SEM	9.45	35.7	3.520	9.48	1.330	13.6
Median	16.50	144.0	13.500	38.00	4.950	30.0
Variance	536.0	6370.0	49.600	449.00	7.040	1120.0
StDev	23.10	79.8	7.050	21.20	2.650	33.4
Maximum	48.00	147.0	15.000	40.00	5.800	62.0
Minimum	9e-03	5e-03	0.003	3e-03	0.005	4e-03
Range	48.00	147.0	15.000	40.00	5.790	62.0
Skewness	0.17	-0.6	-1.920	-0.60	-1.830	1e-03
Kurtosis	-2.89	-3.3	3.730	-3.32	3.450	-3.3
CV	112.00	91.3	67.100	91.30	67.600	110.0

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 22. Analysis of B in Sludge A Samples

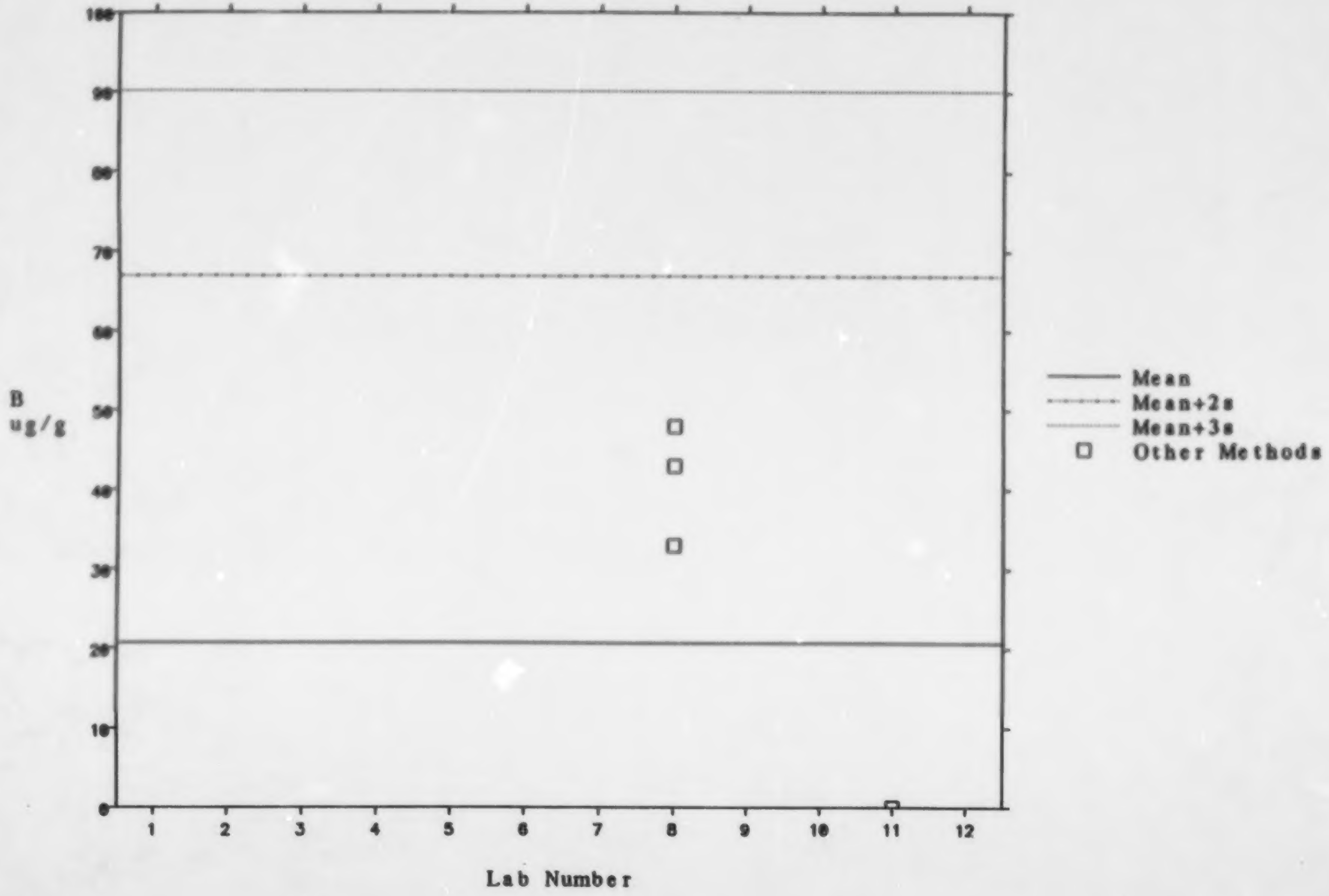


Figure 23. Analysis of B in Sludge B

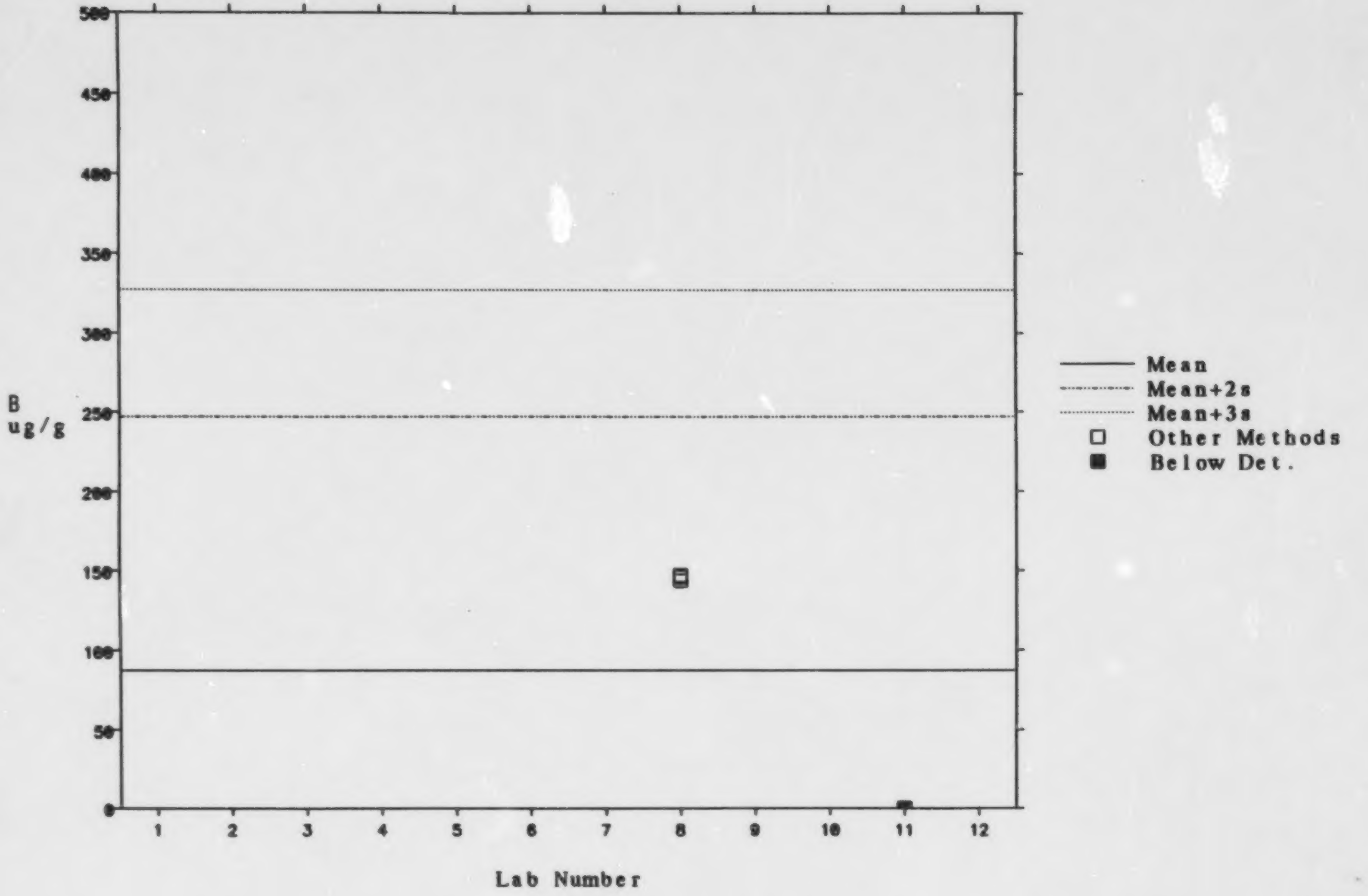


Figure 24. Analysis of B in Sludge C

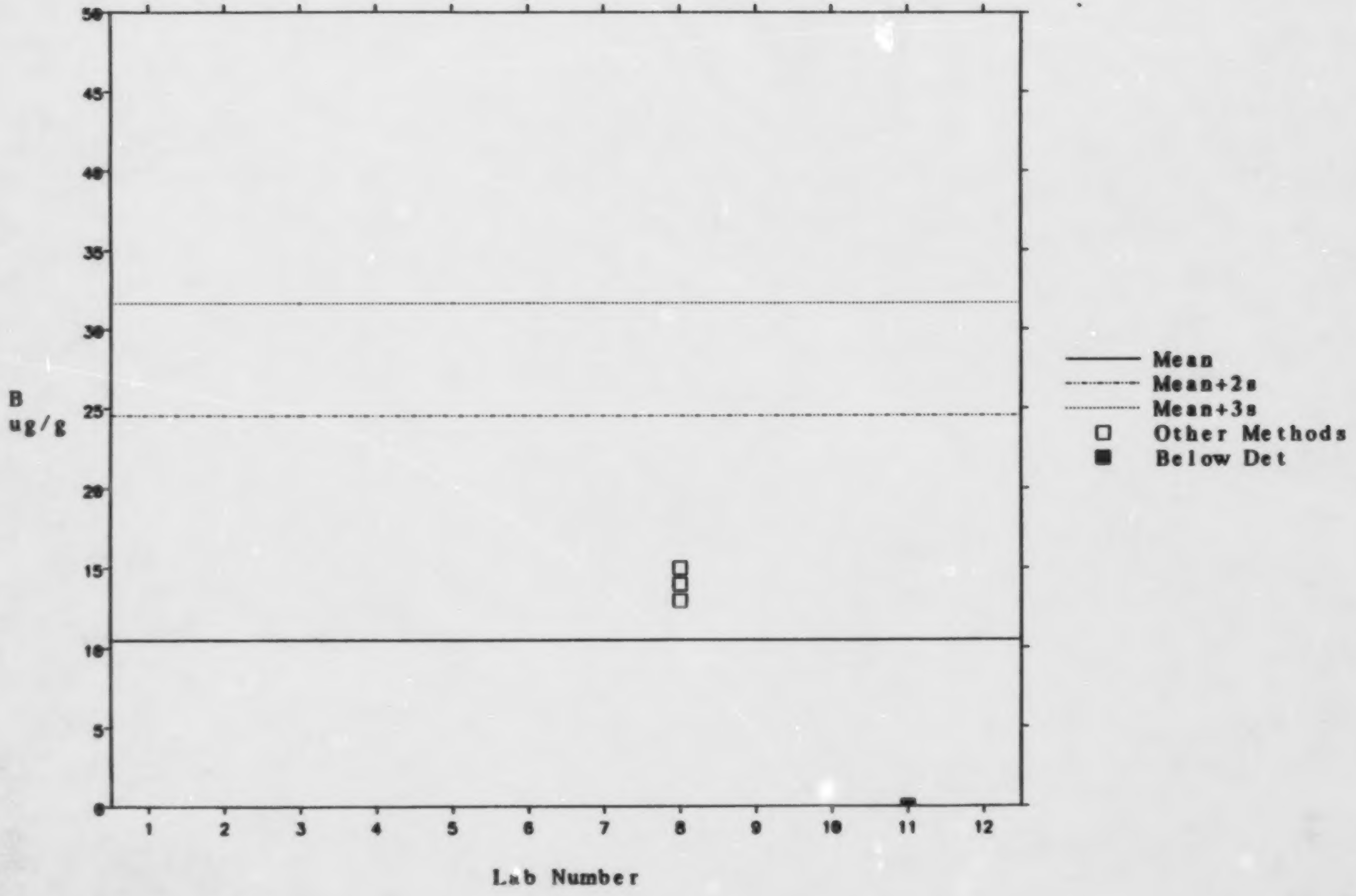


Figure 25. Analysis of B in Sludge D

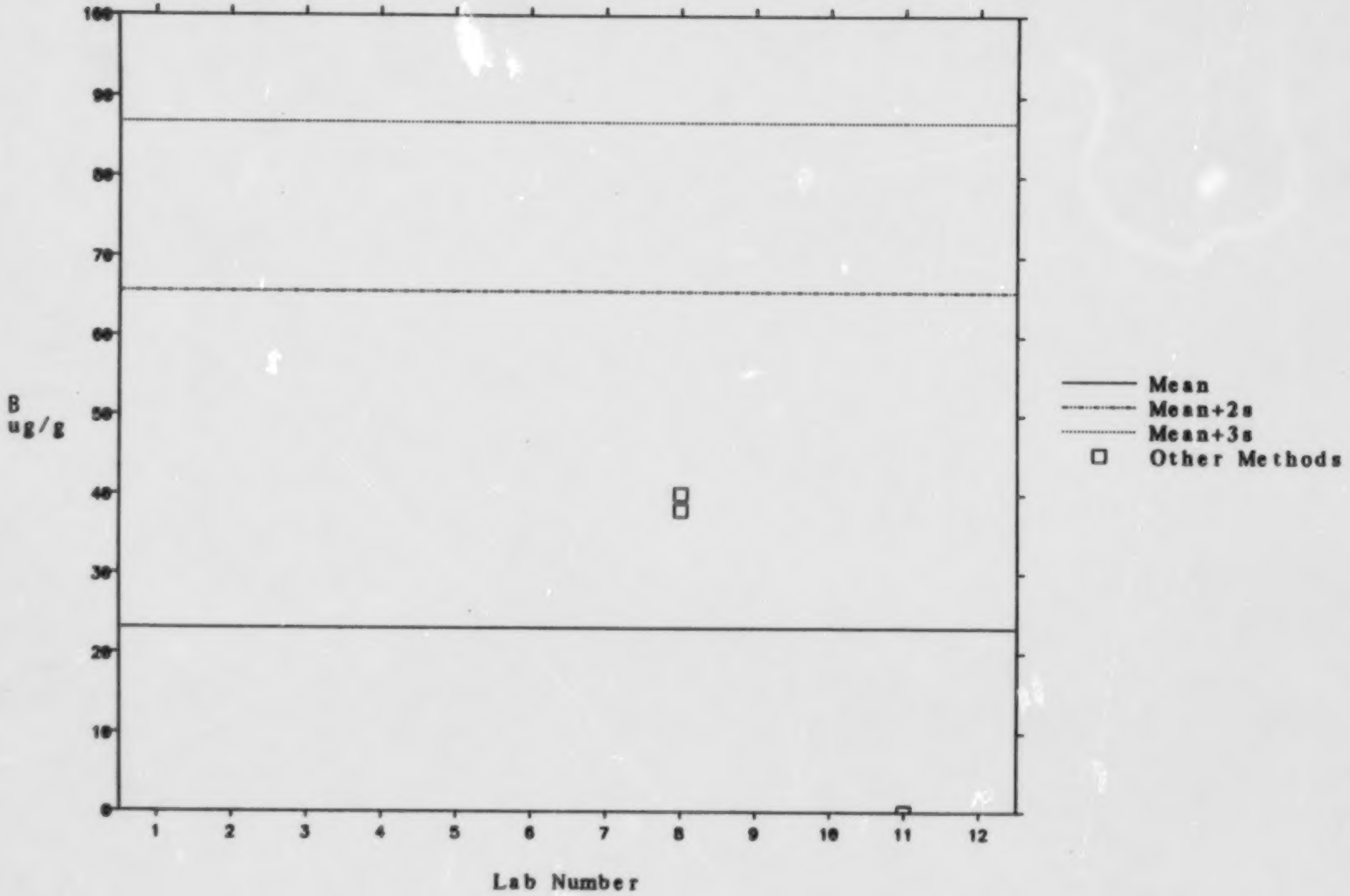


Figure 26. Analysis of B in Sludge E

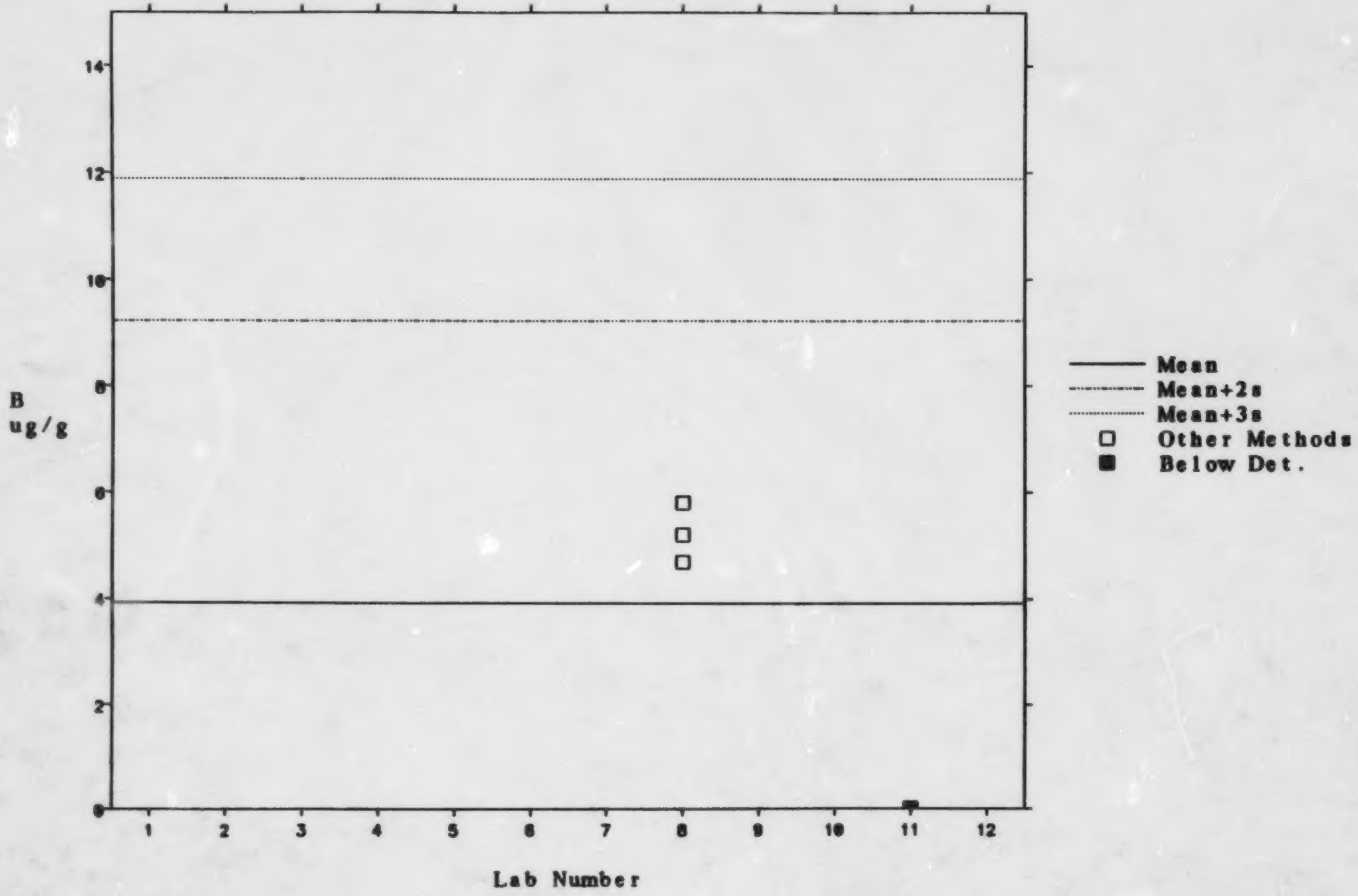


Figure 27. Analysis of B in Sludge F

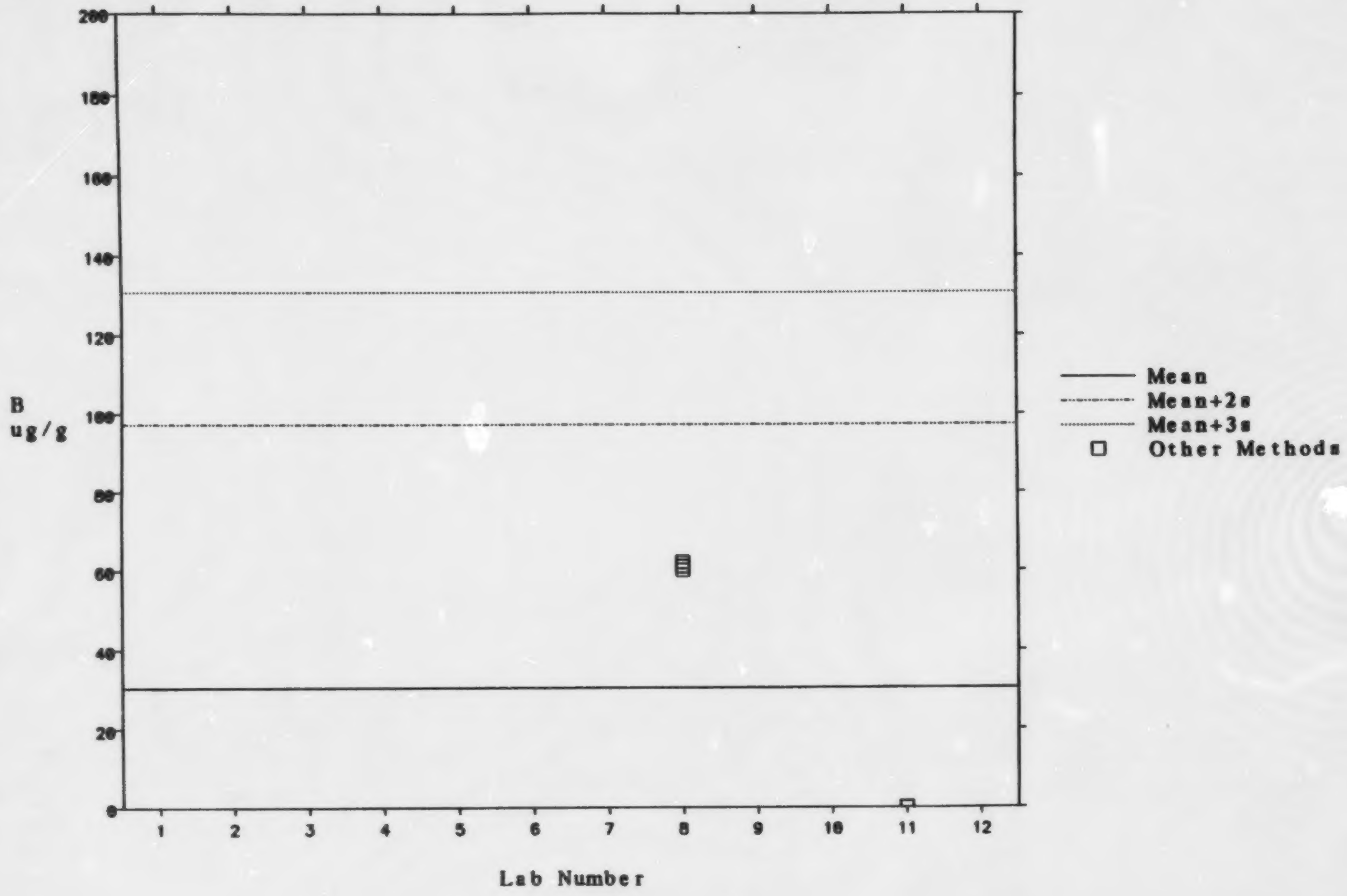


Figure 28. Mean and Confidence Intervals for Ba in an ICP Standard

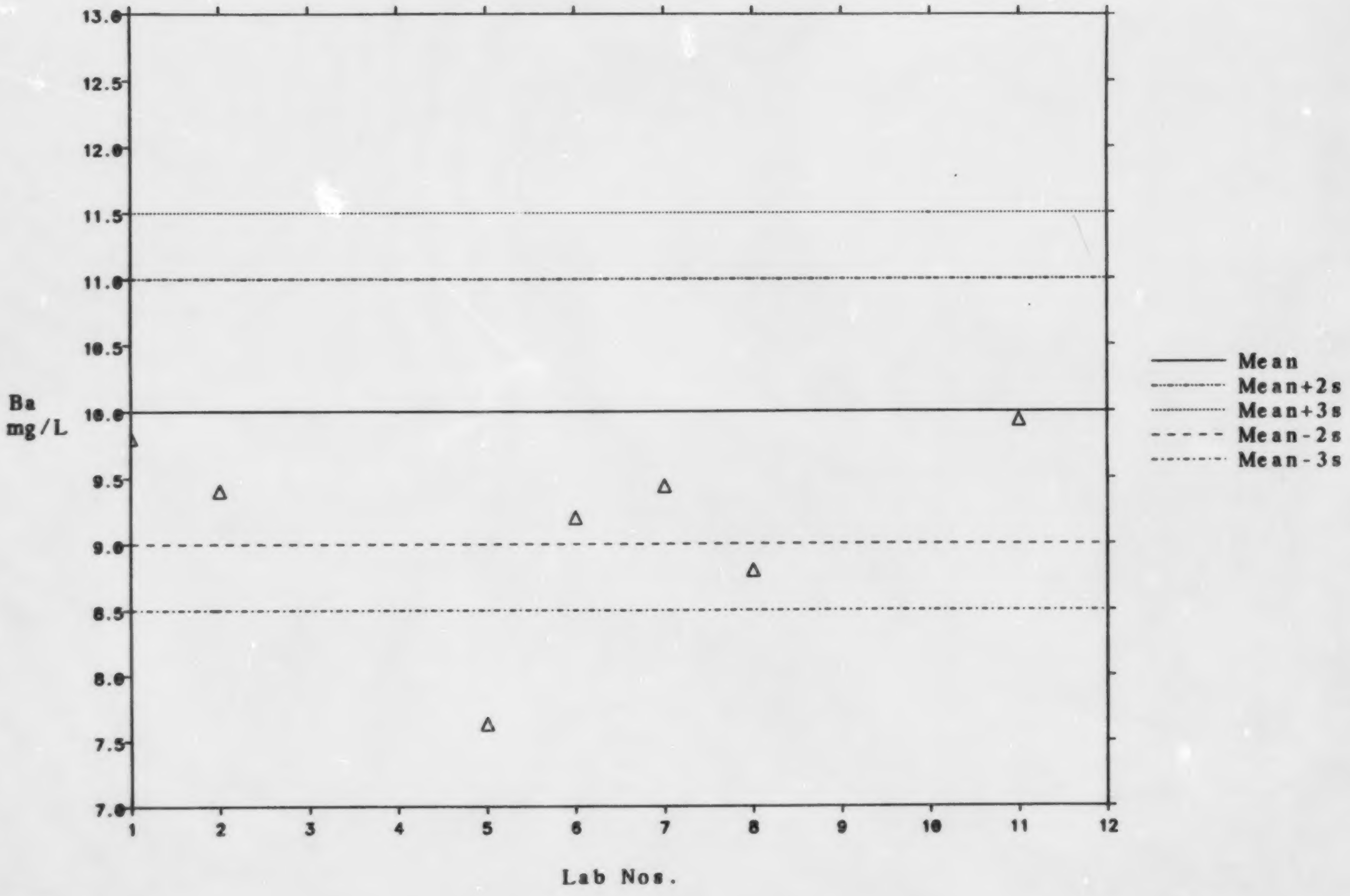


Figure 29. Mean and Confidence Intervals for Ba in Digestate 3

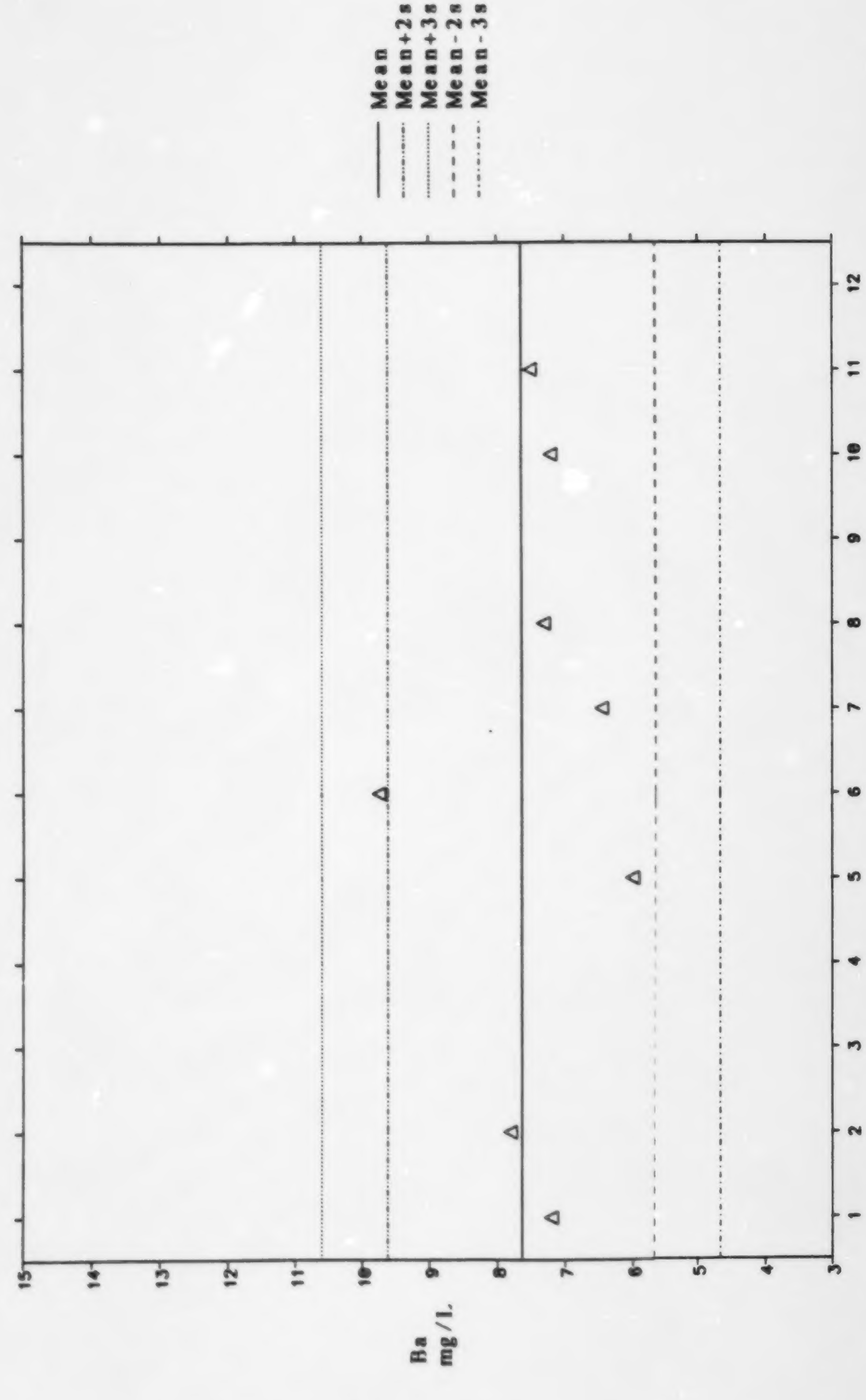


Figure 30. Mean and Confidence Intervals for Ba in Digestate 4

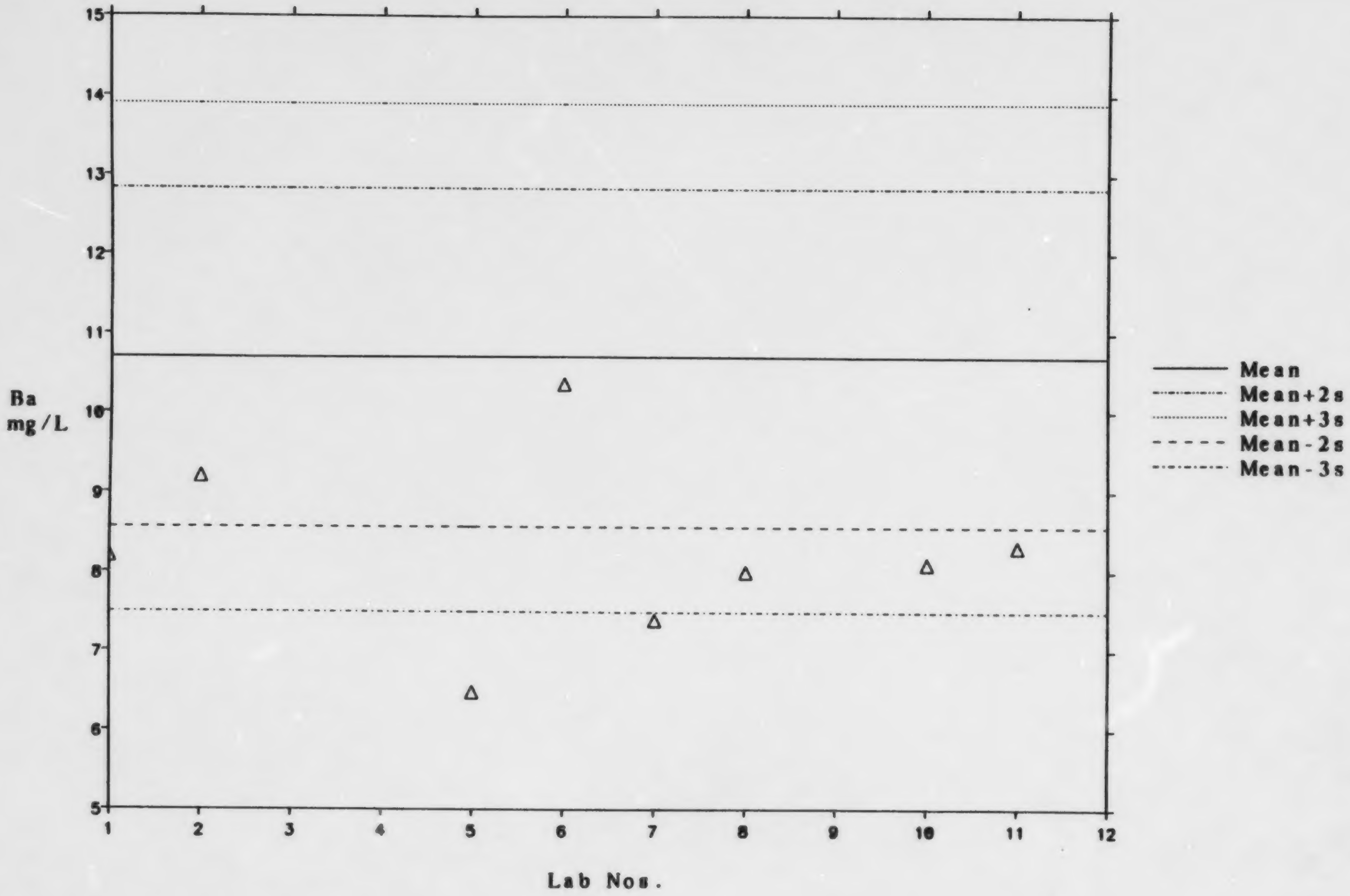


Figure 31. Mean and Confidence Intervals for Ba in Standard A

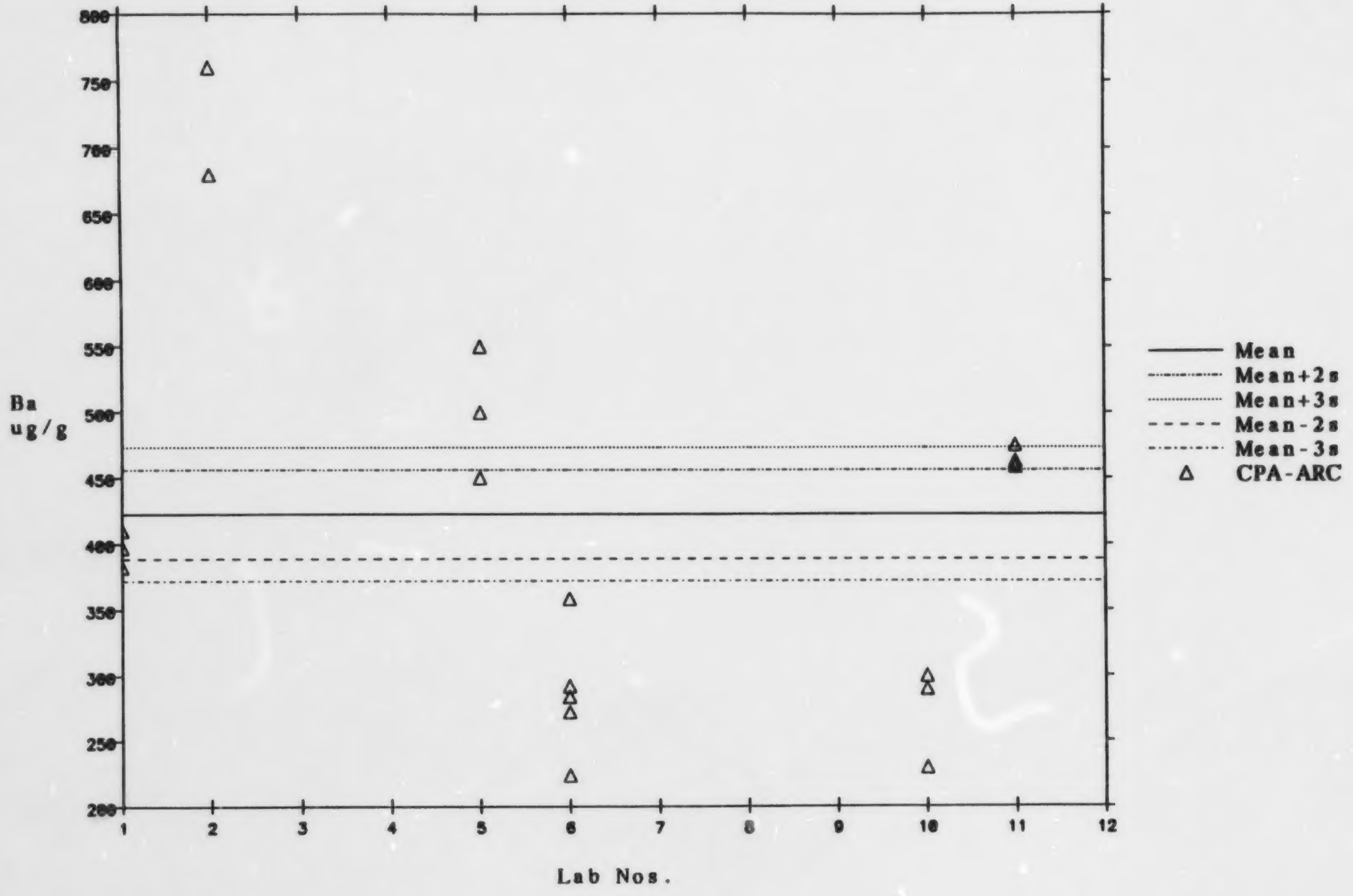
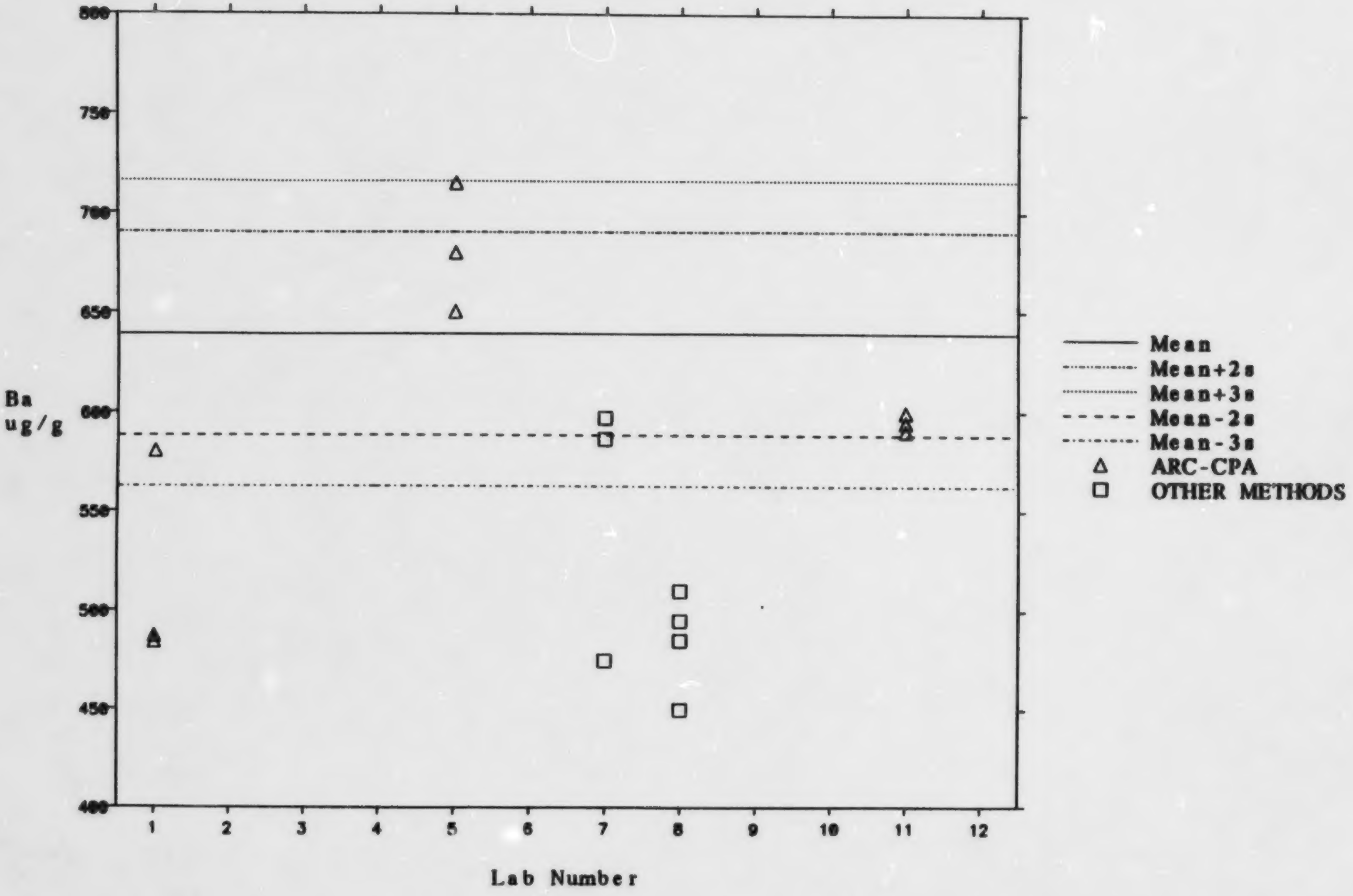


Figure 32. Mean and Confidence Intervals for Ba in Standard B



The statistical data for Ba in sludges A to F are shown in Table 5, and their control charts in Figures 33 to 38. Table 5 shows that results (above detection) were reported for all sludges, with number of analyses varying from 10 to 41 samples. The mean Ba values ranged from 9.9 mg/kg for sludge E to 900 for sludge B. The coefficient of variation of the sludges varied from 21 to 216% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 33 to 38 indicate that all the reported Ba data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Ba data shows good agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed and the Ba concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.1.5 Be

Three labs reported Be in the ICP standard (Figure 39), all but one lab were within the control limits. Four labs reported Be for the digestate samples (Figures 40 and 41) with all the data falling within the control limits. Four labs reported Be for the standard A and B samples (Figures 42 and 43). Figures 42 and 43 show that 2 labs reported data within Standards A and B warning limits and the other 2 were outside those limits. These figures also show that the ARC-CPA method data was mostly within the control limits, while the one other method was outside these limits.

The statistical data for Be in sludges A to F are shown in Table 6, and their control charts in Figures 44 to 49. Table 6 shows that results (above detection) were reported for all sludges, with number of analyses varying from 1 to 10 samples. The mean Be values ranged from 0.1 mg/kg for sludge C to 1.8 for sludge F. The coefficient of variation of the sludges varied from 0 (2 points only) to 128%.

Table 5. Statistical Parameters for Ba (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	36	41	41	38	10.00	34
Sum	8750	36900	24800	19500	98.90	28500
Mean	243	900	605	514	9.89	838
SEM	42	29	78	24	6.75	83
Median	136	940	420	495	2.40	780
Variance	63500	33900	248000	22100	456.00	233000
StDev	252	184	498	149	21.40	483
Maximum	930	1180	1790	750	70.00	1800
Minimum	69	500	140	235	0.40	77
Range	861	680	1650	515	69.60	1720
Skewness	2	-1	2	3e-02	3.04	3e-01
Kurtosis	2	7e-02	2	-1	9.41	-7e-01
CV	104	21	82	29	216.00	58

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 33. Analysis of Ba in Sludge A

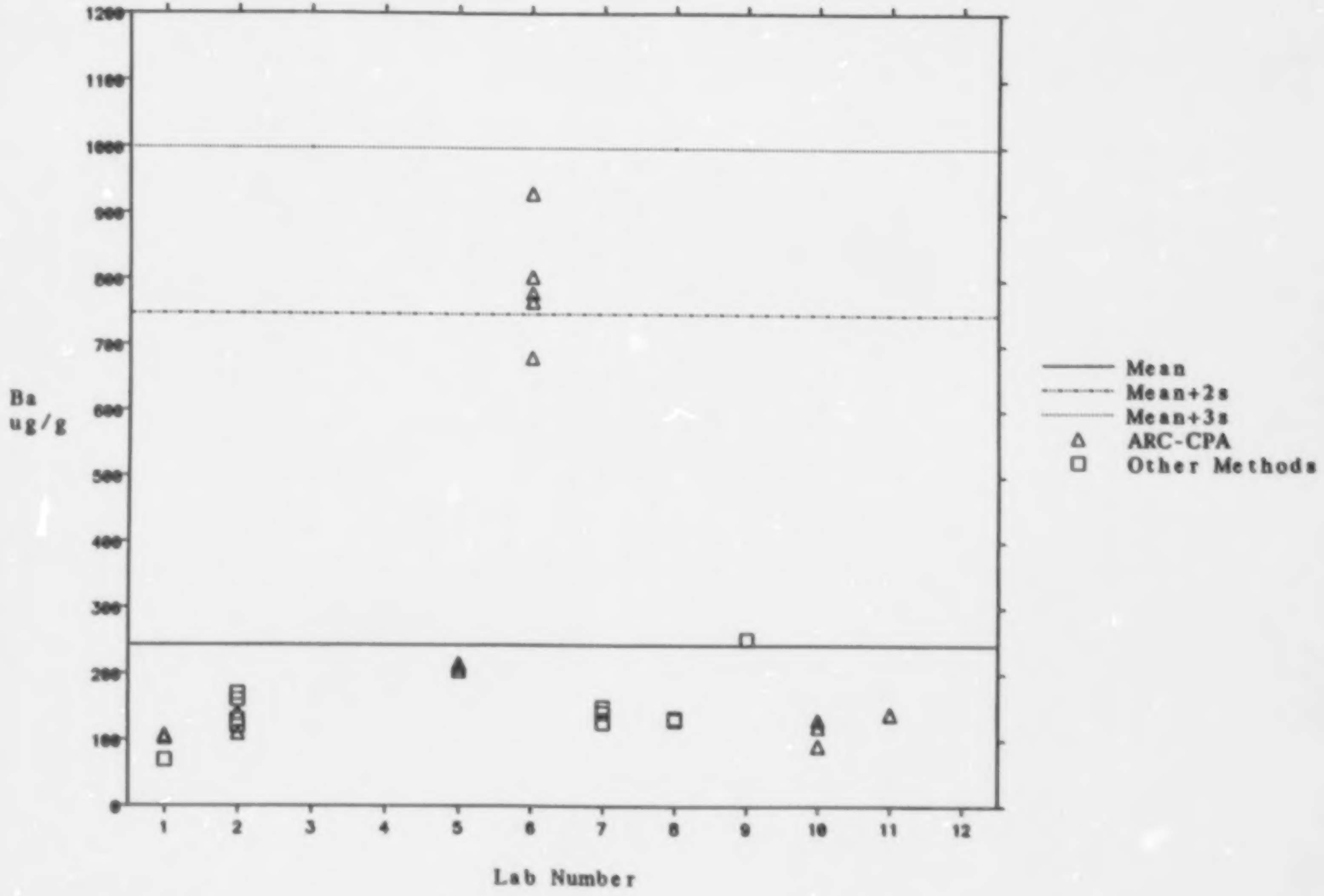


Figure 34. Analysis of Ba in Sludge B

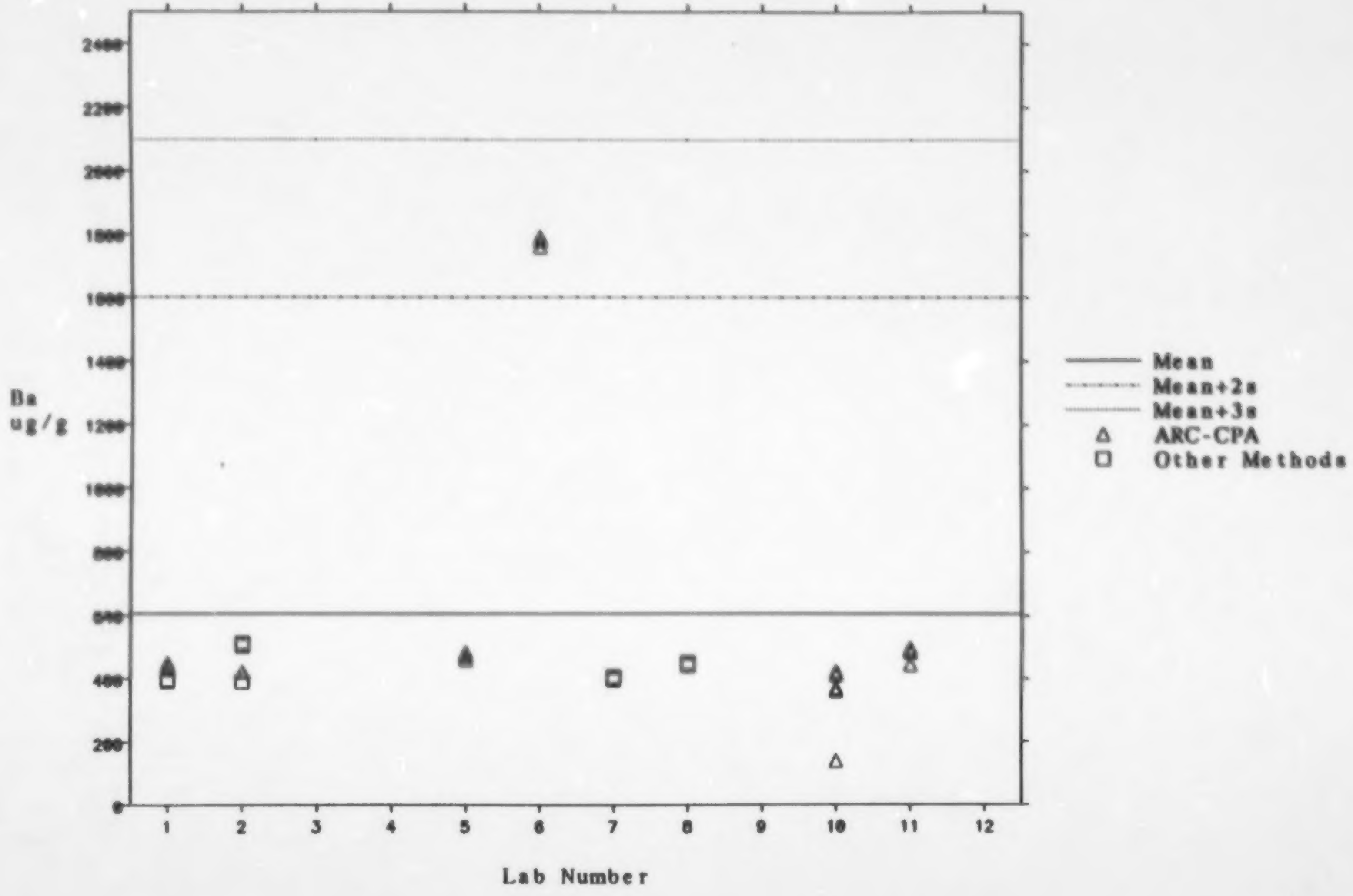


Figure 35. Analysis of Ba in Sludge C

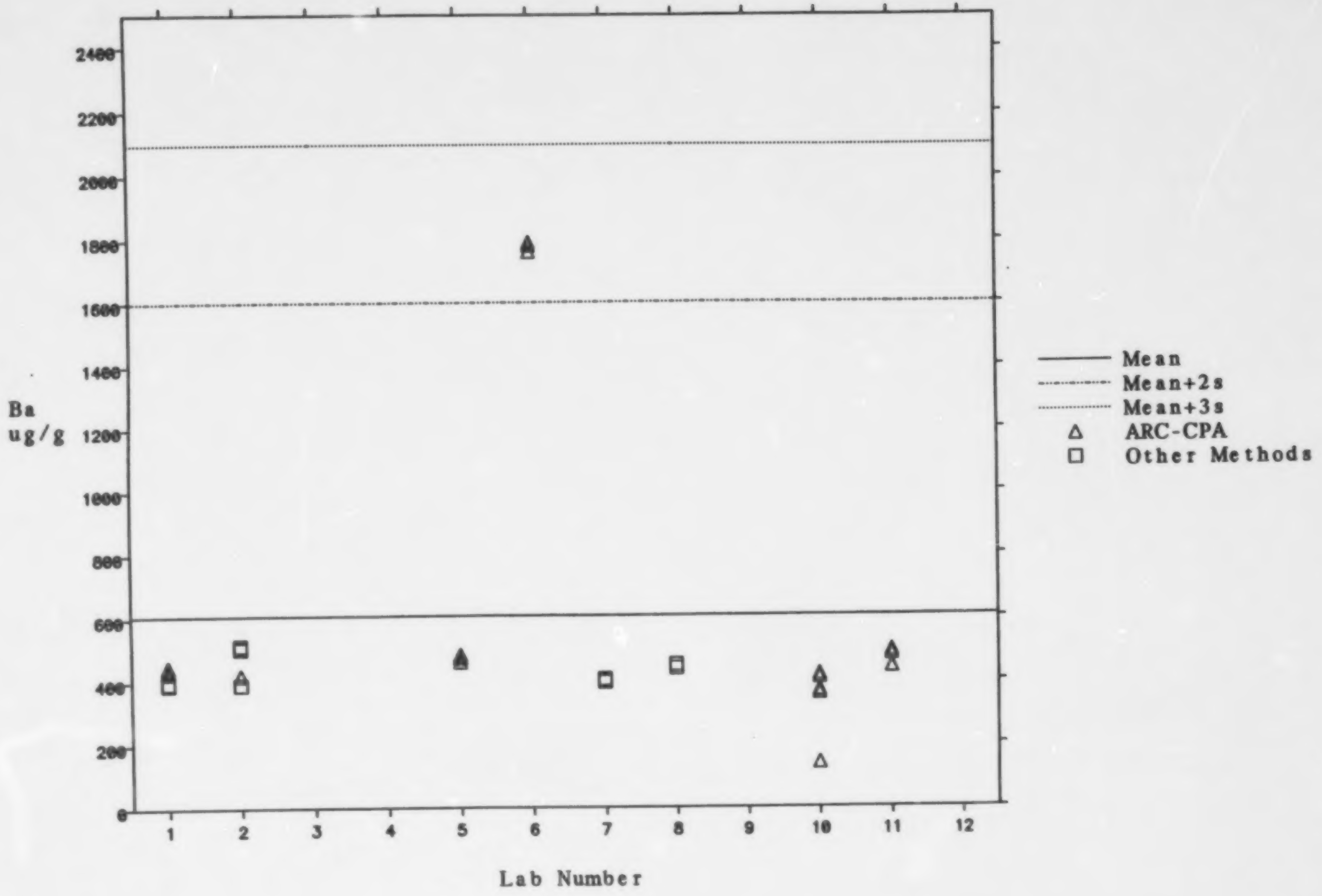


Figure 36. Analysis of Ba in Sludge D

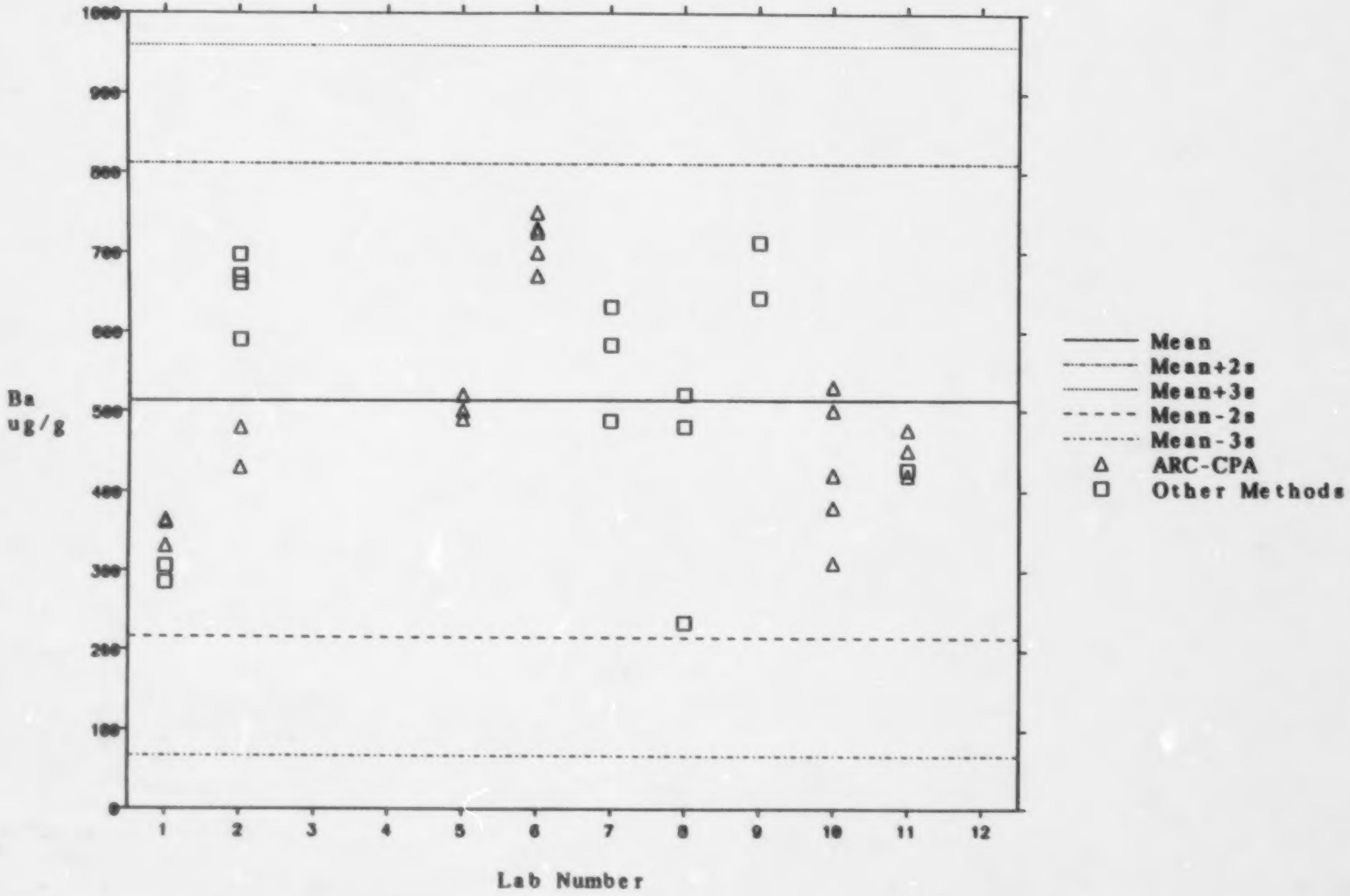


Figure 37. Analysis of Ba in Sludge E

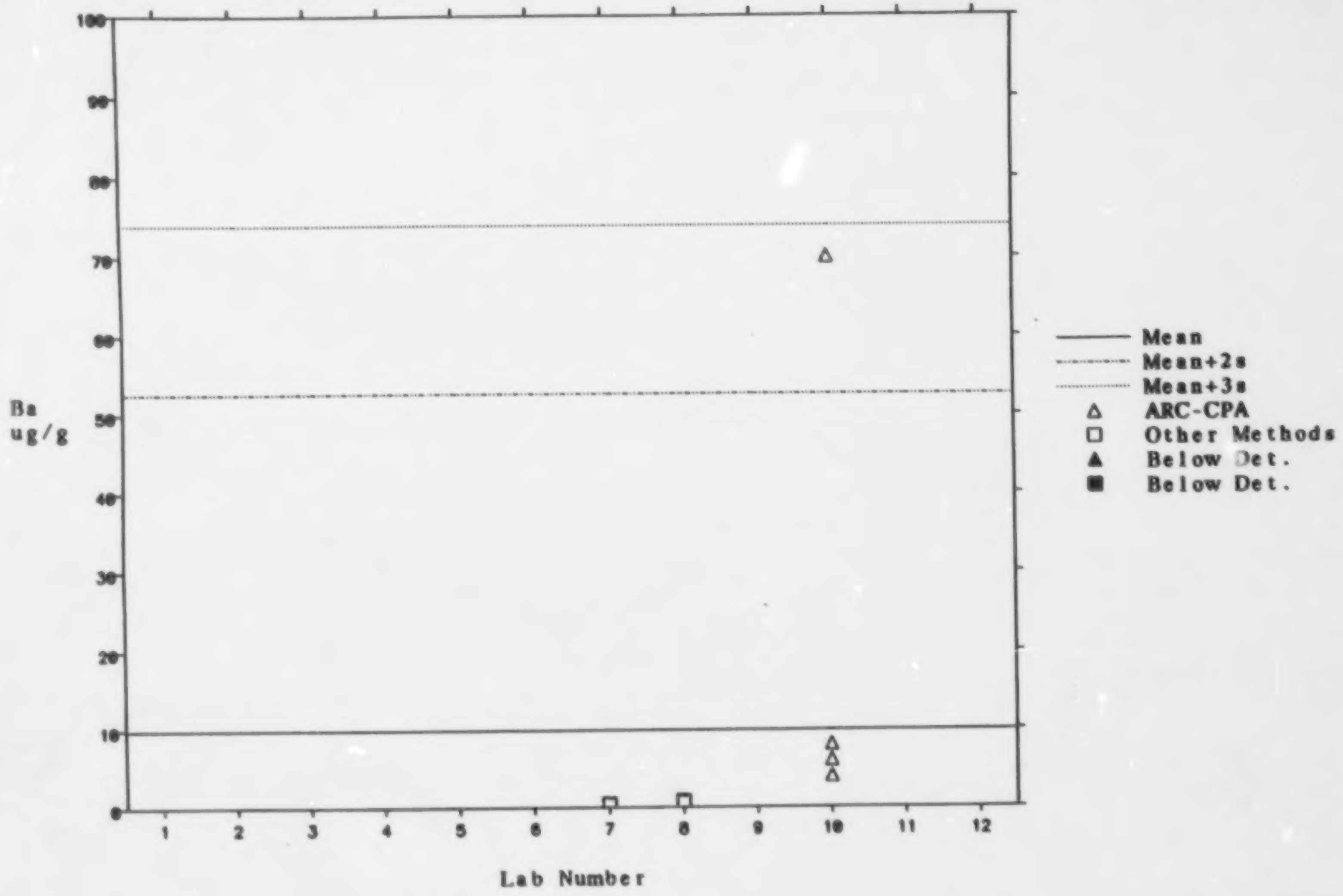


Figure 38. Analysis of Ba in Sludge F

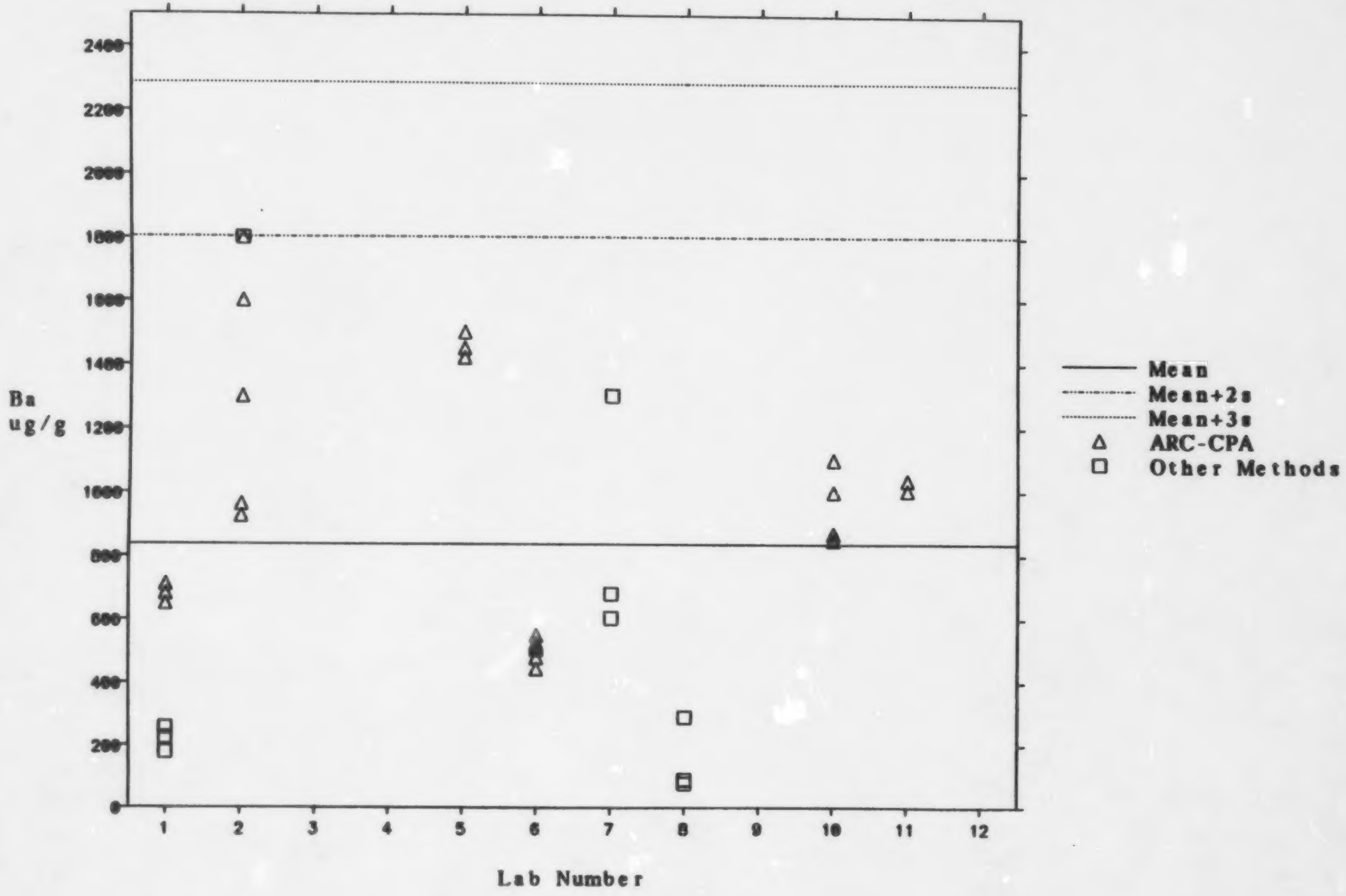


Figure 39. Mean and Confidence Intervals for Be in an ICP Standard

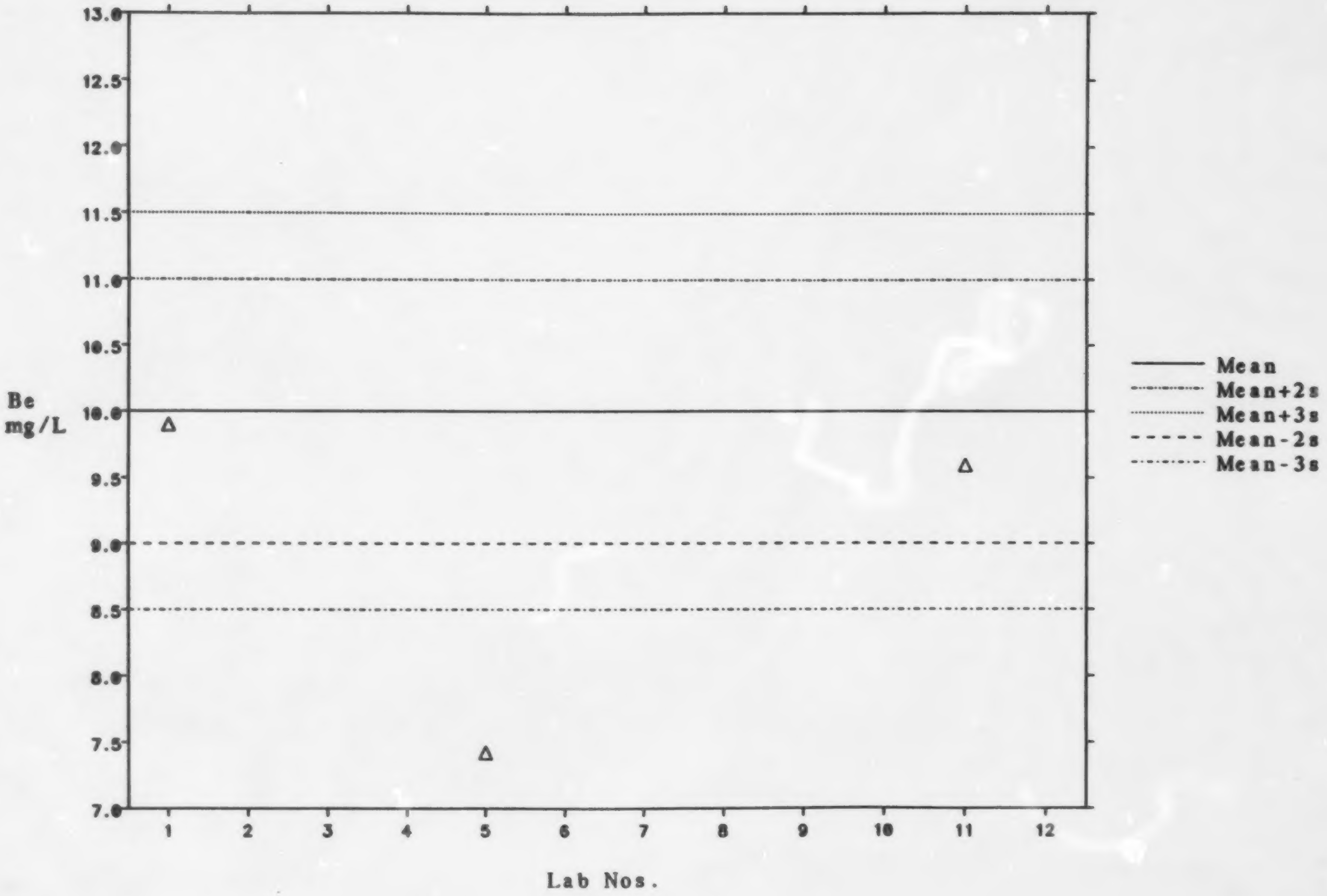


Figure 40. Mean and Confidence Intervals for Be in Digestate 3

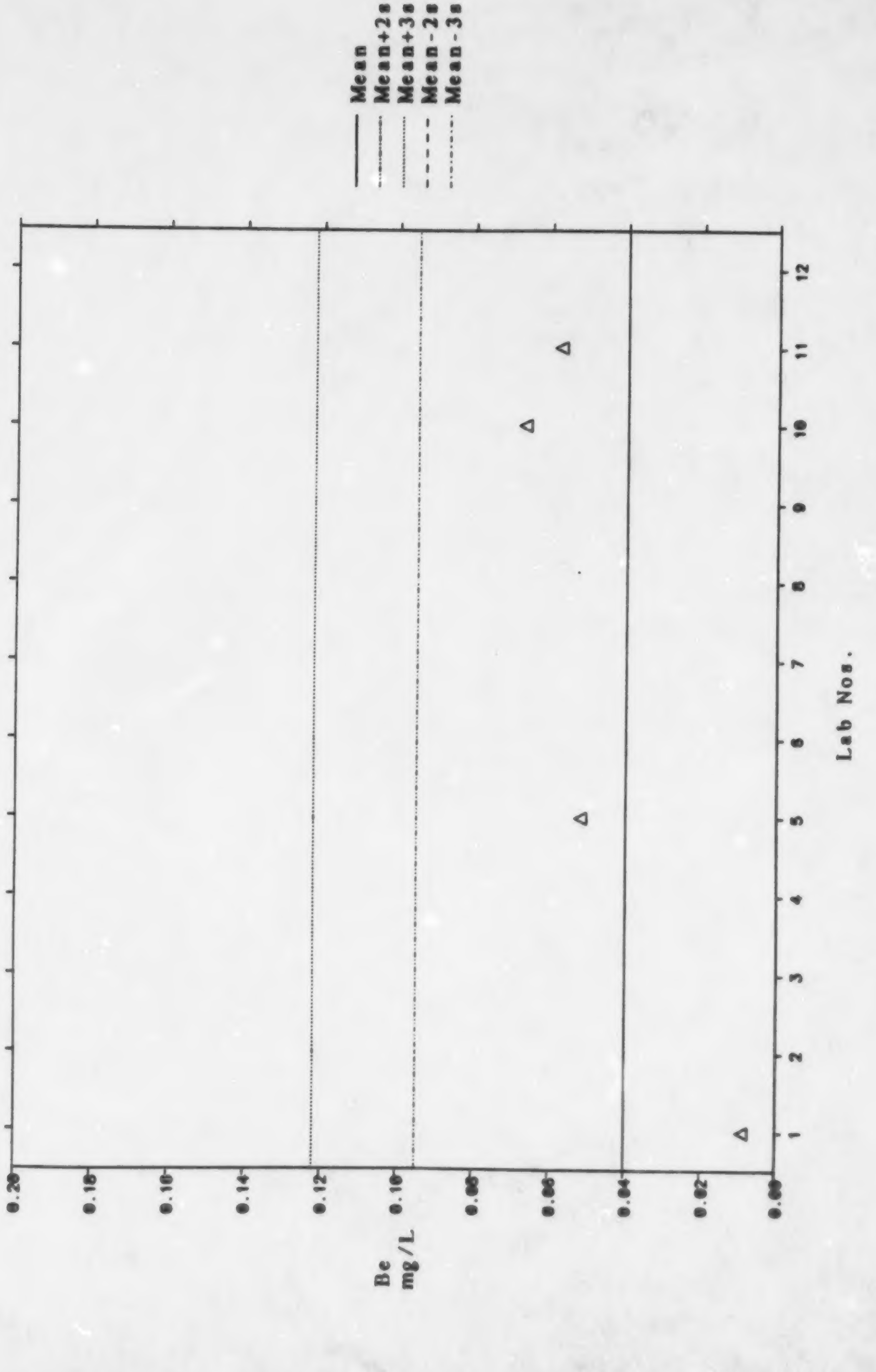


Figure 41. Mean and Confidence Intervals for Be in Digestate 4

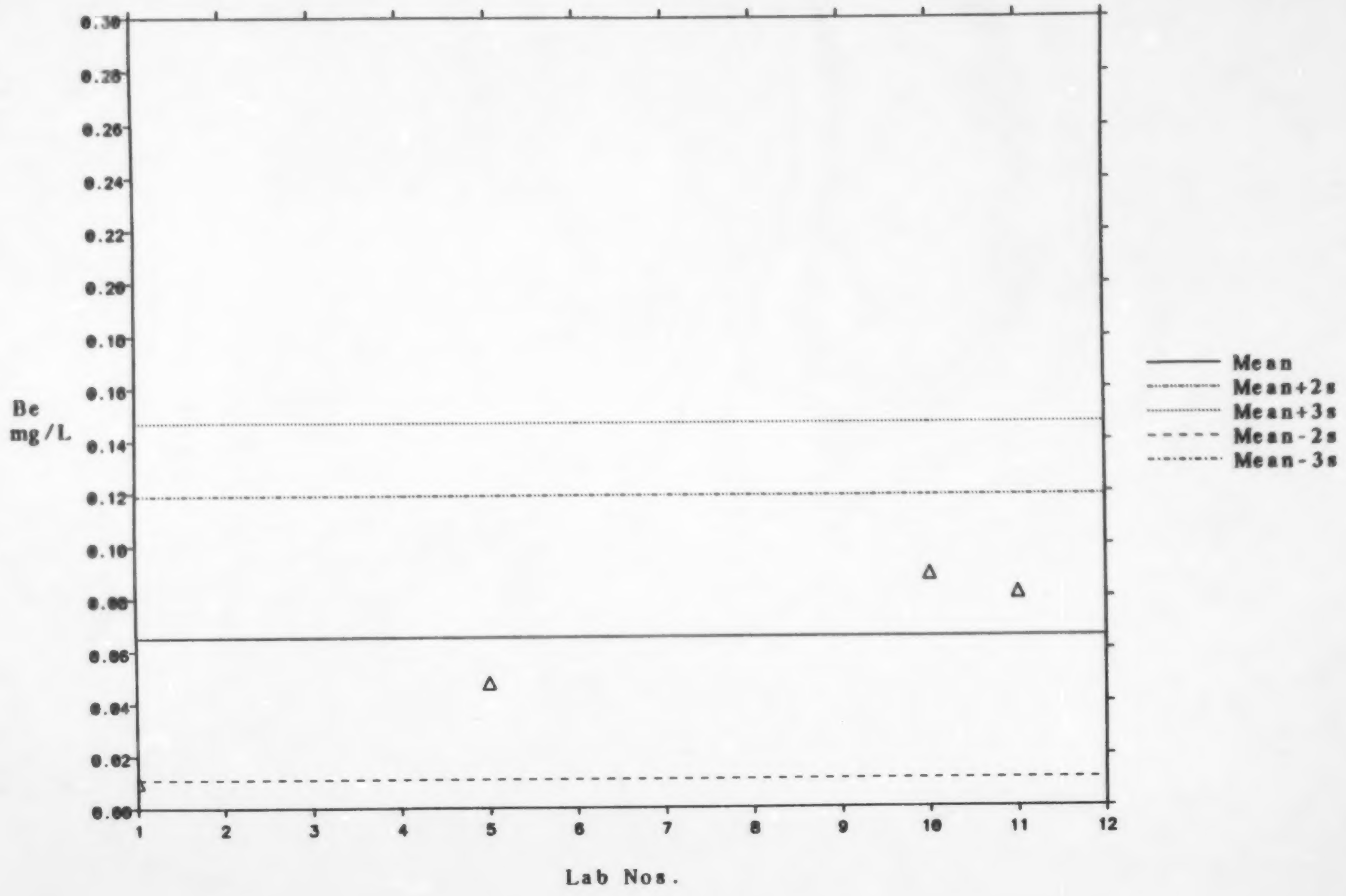


Figure 42. Mean and Confidence Intervals for Be in Standard A

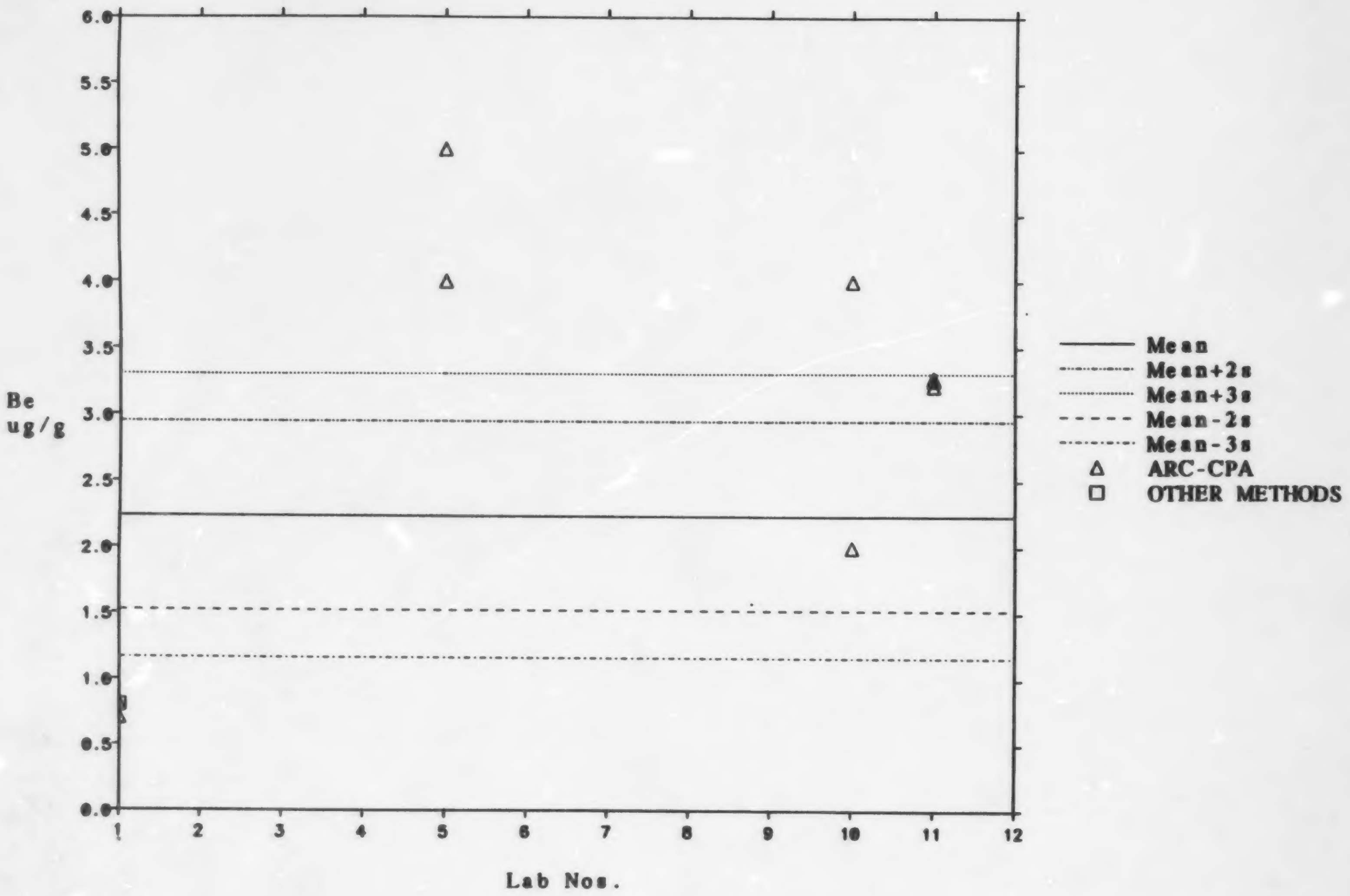


Figure 43. Mean and Confidence Intervals for Be in Standard B

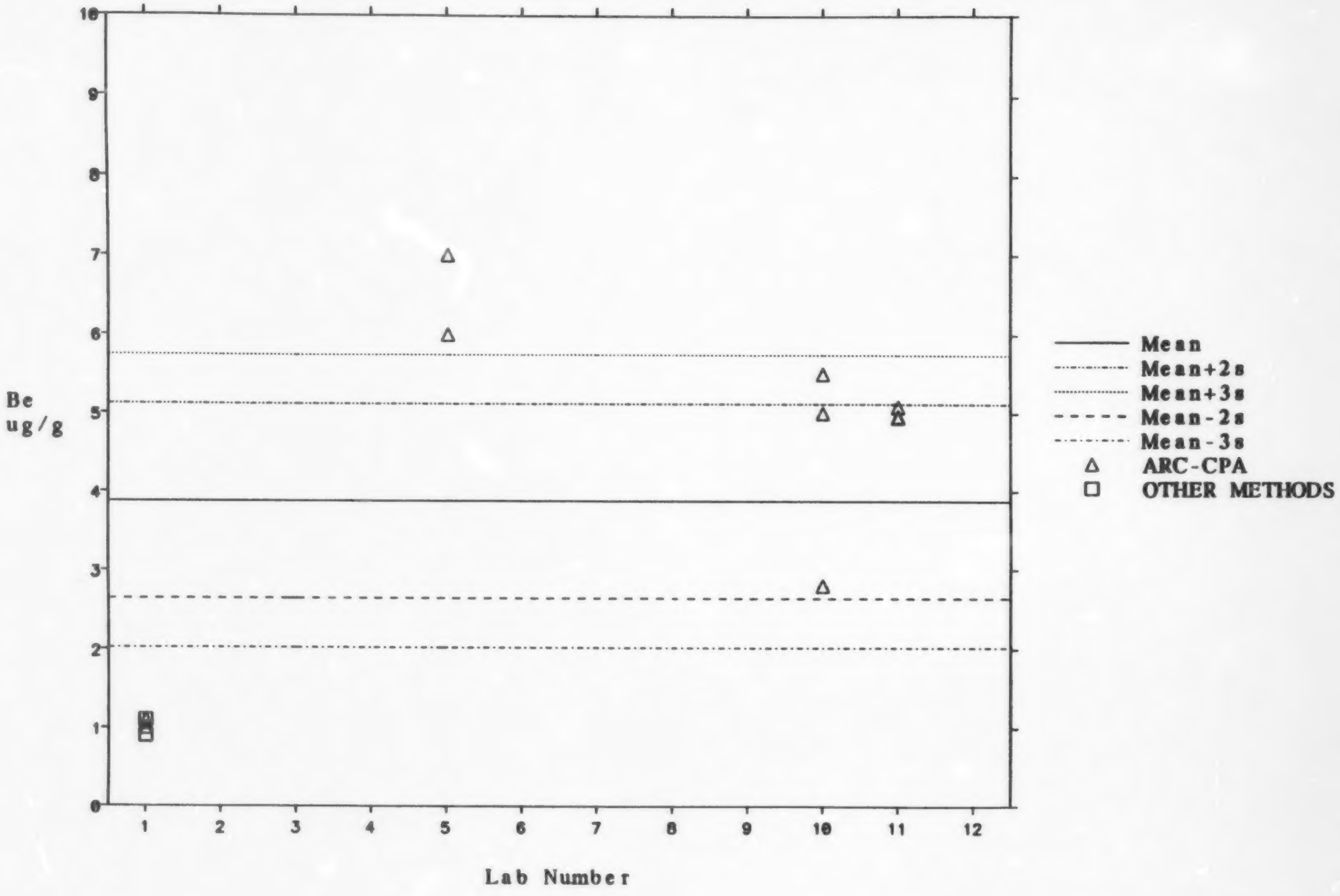


Table 6. Statistical Parameters for Be (mg/Kg) in Sludges A-F

Statistic	Sludge					
	A	B	C	D	E	F
Count	10.000	7.00	1.0	5.000	2.0	6.000
Sum	3.930	7.32	0.1	1.430	0.2	10.700
Mean	0.393	1.05	0.1	0.286	0.1	1.790
SEM	0.092	0.51		0.109	0.0	0.551
Median	0.342	0.83	0.1	0.138	0.1	2.000
Variance	0.084	1.80		0.060	0.0	1.820
StDev	0.290	1.34		0.244	0.0	1.350
Maximum	1.000	4.00	0.1	0.600	0.1	3.000
Minimum	0.100	0.20	0.1	0.091	0.1	0.349
Range	0.900	3.80	0.0	0.509	0.0	2.650
Skewness	0.927	2.33		0.681		-0.114
Kurtosis	0.686	5.76		-2.760		-3.050
	73.600	128.00		85.200	0.0	75.500

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 44. Analysis of Be in Sludge A

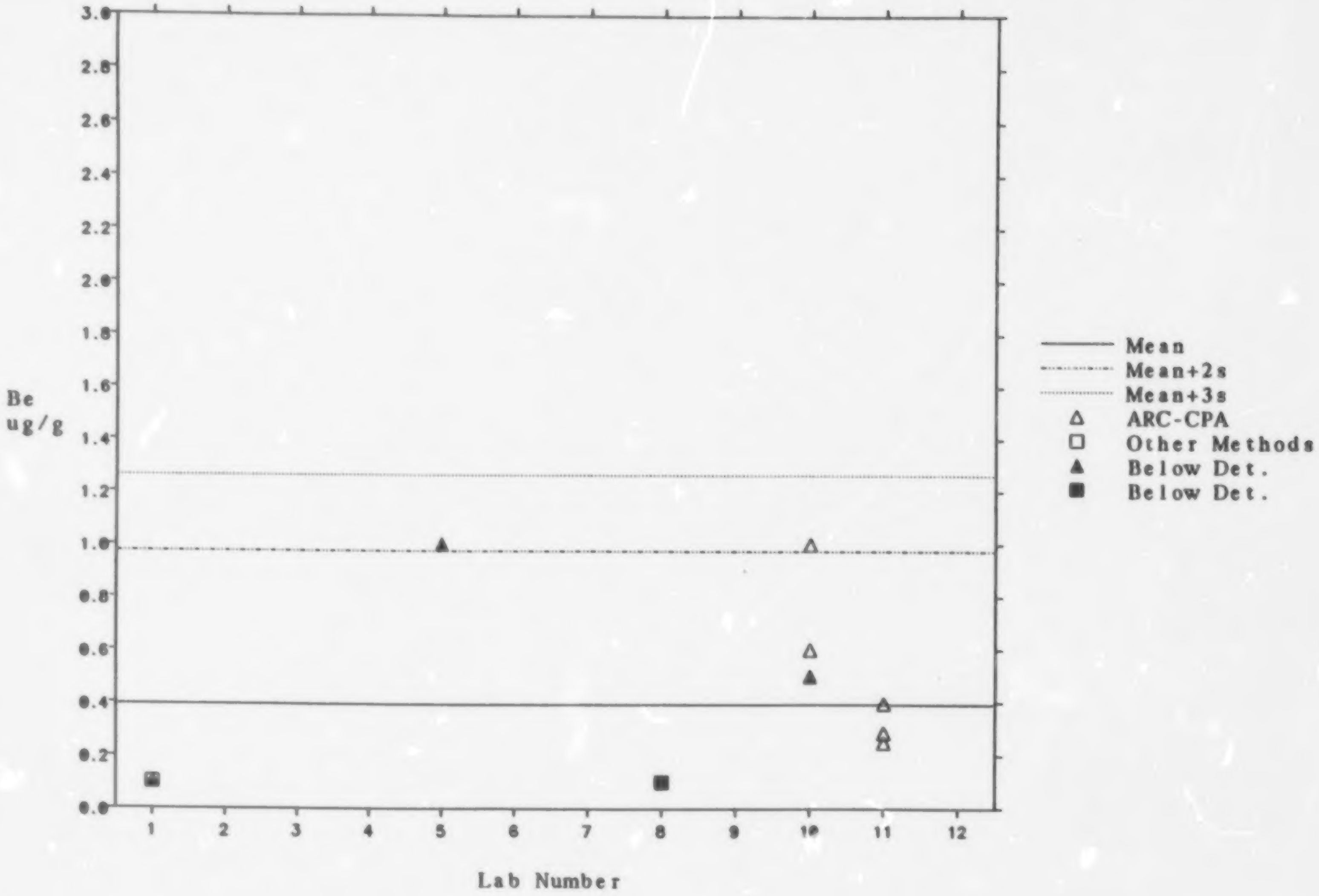


Figure 45. Analysis of Be in Sludge B

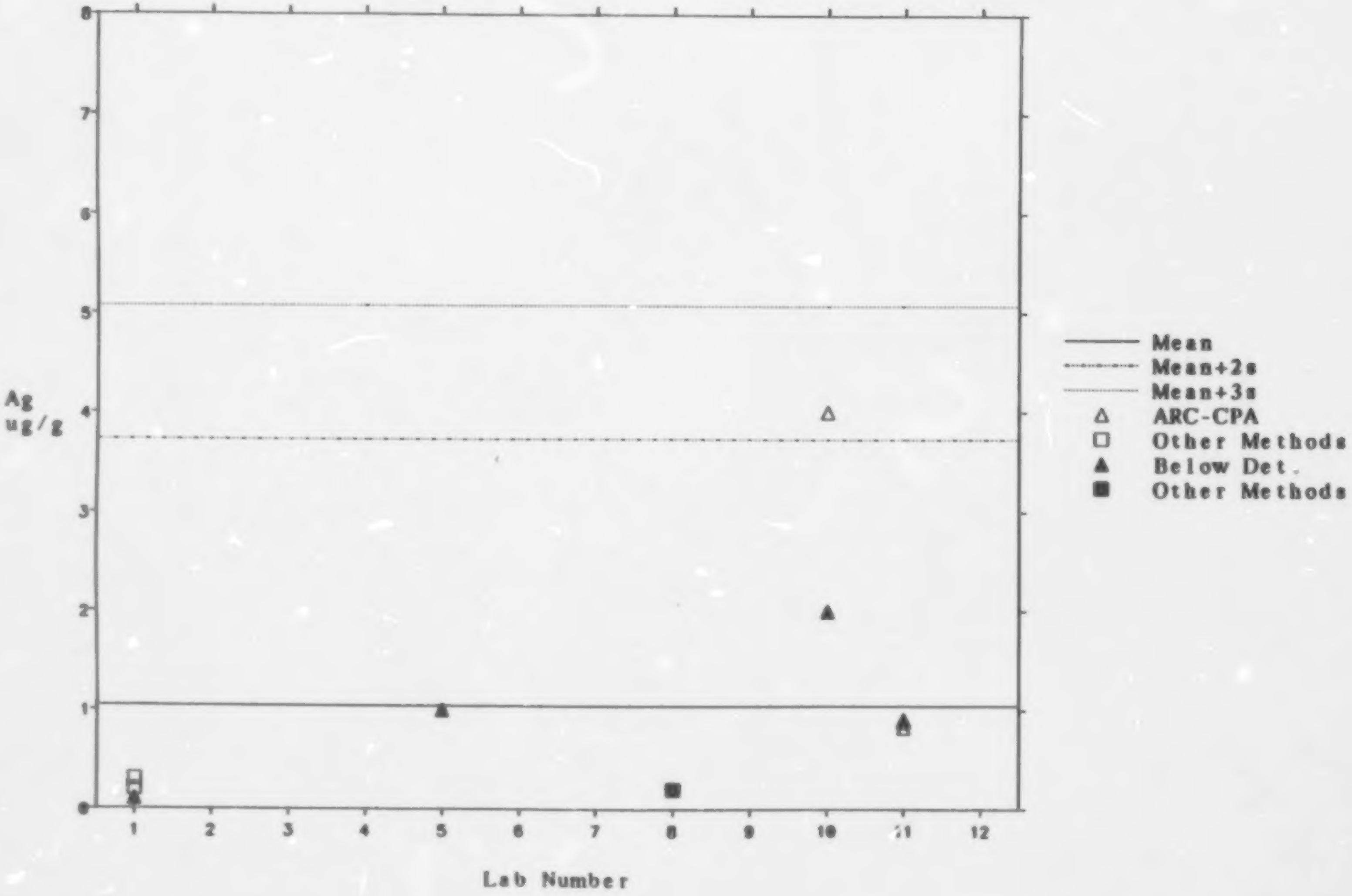


Figure 46. Analysis of Be in Sludge C

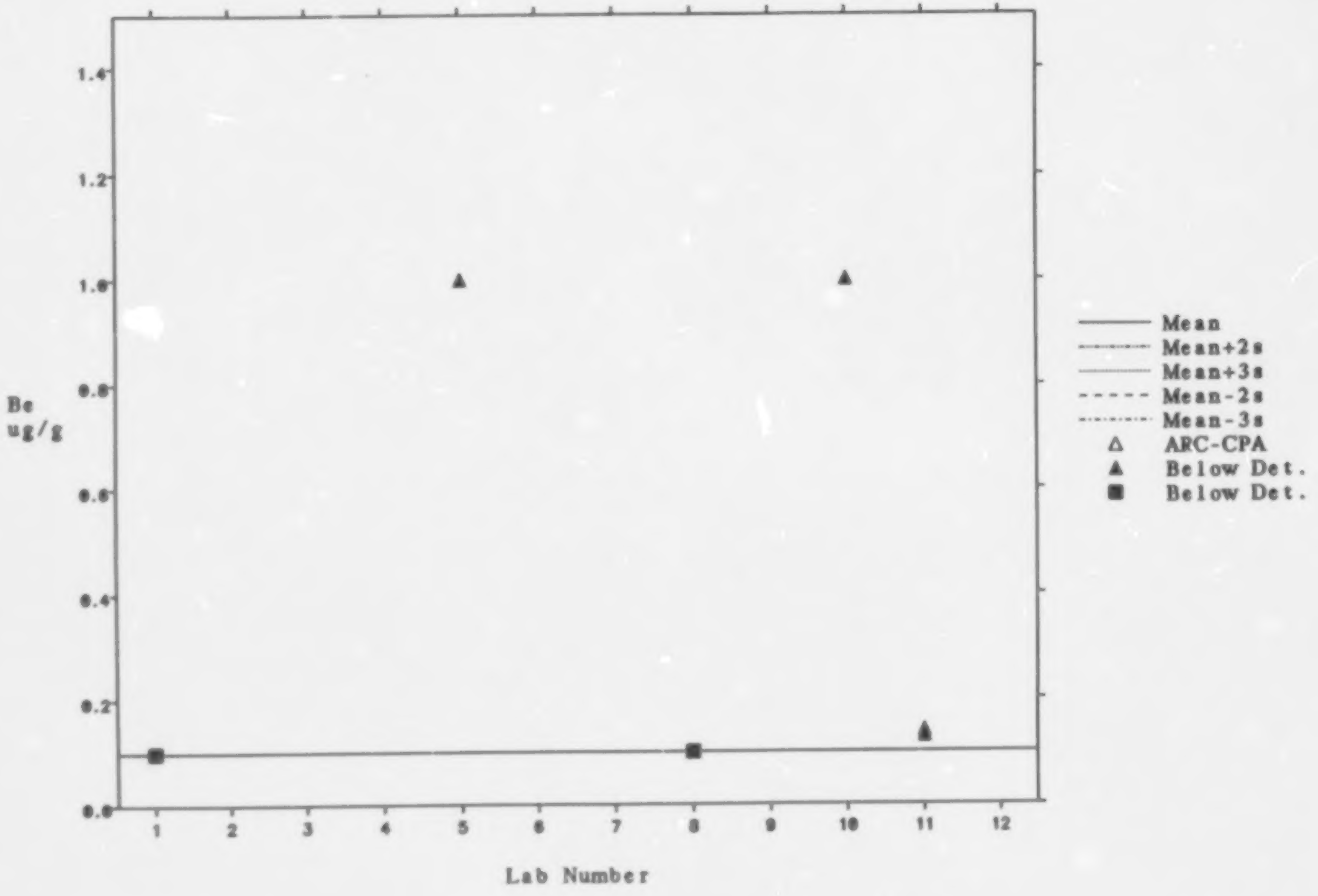


Figure 47. Analysis of Be in Sludge D

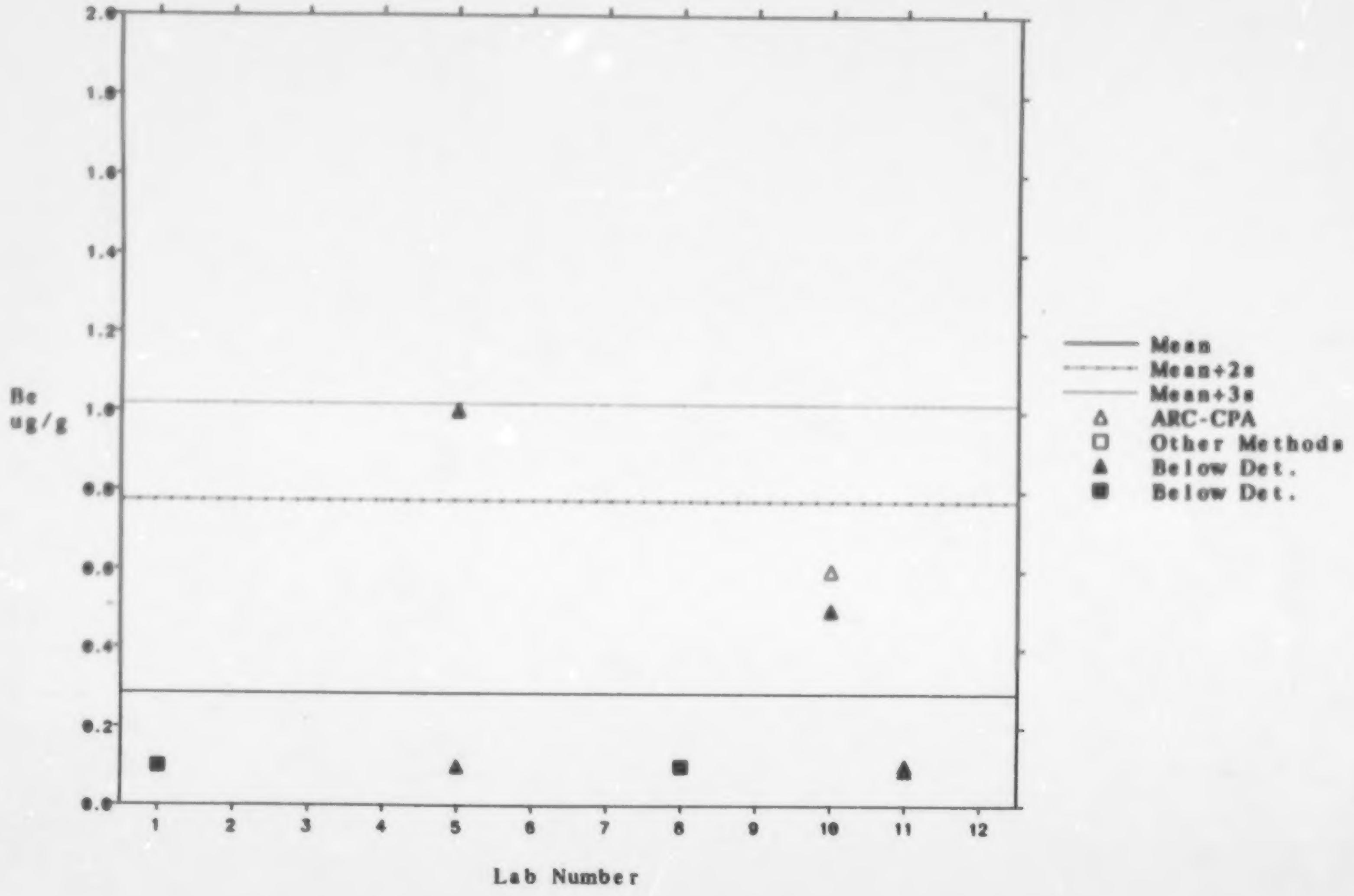


Figure 48. Analysis of Be in Sludge E

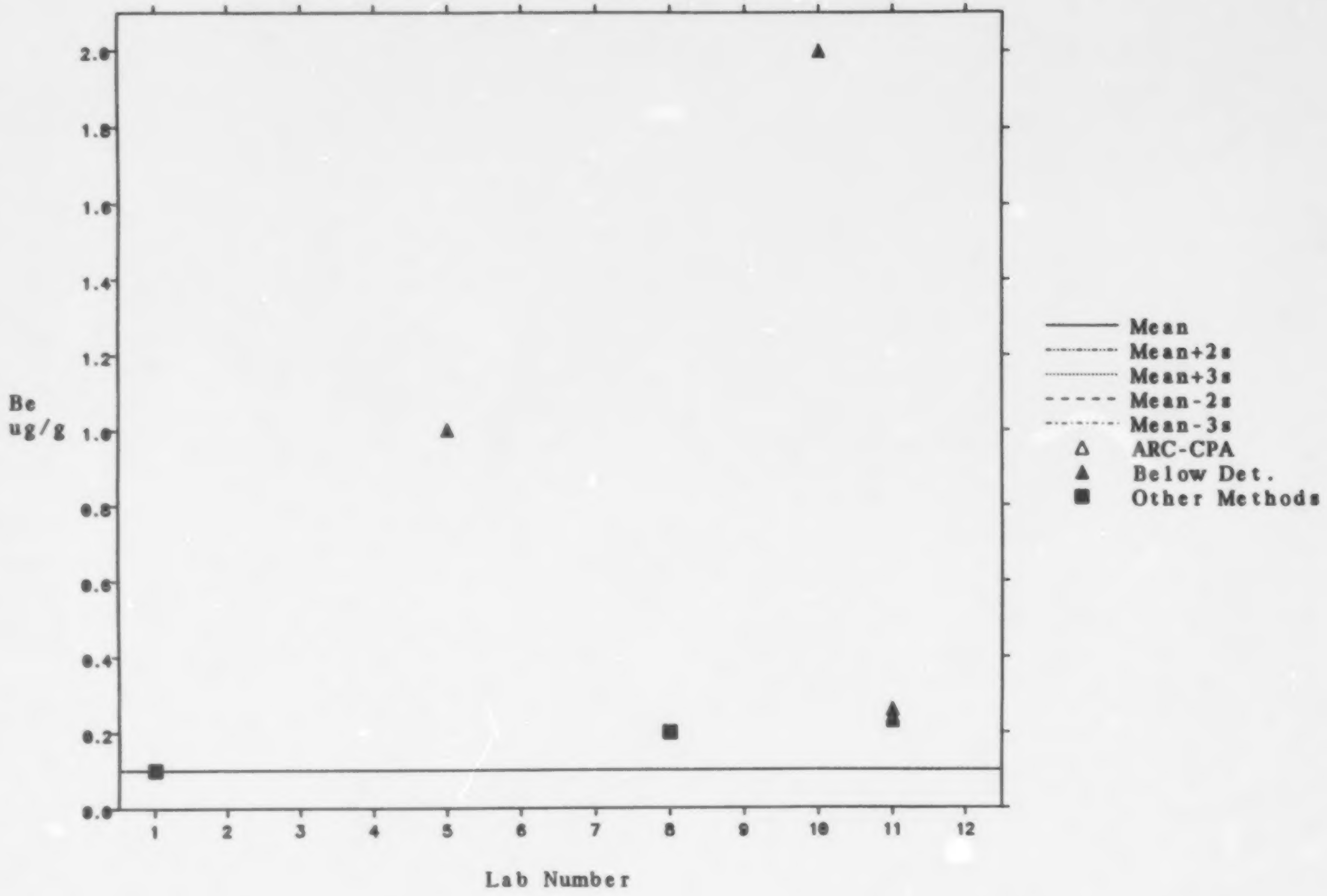
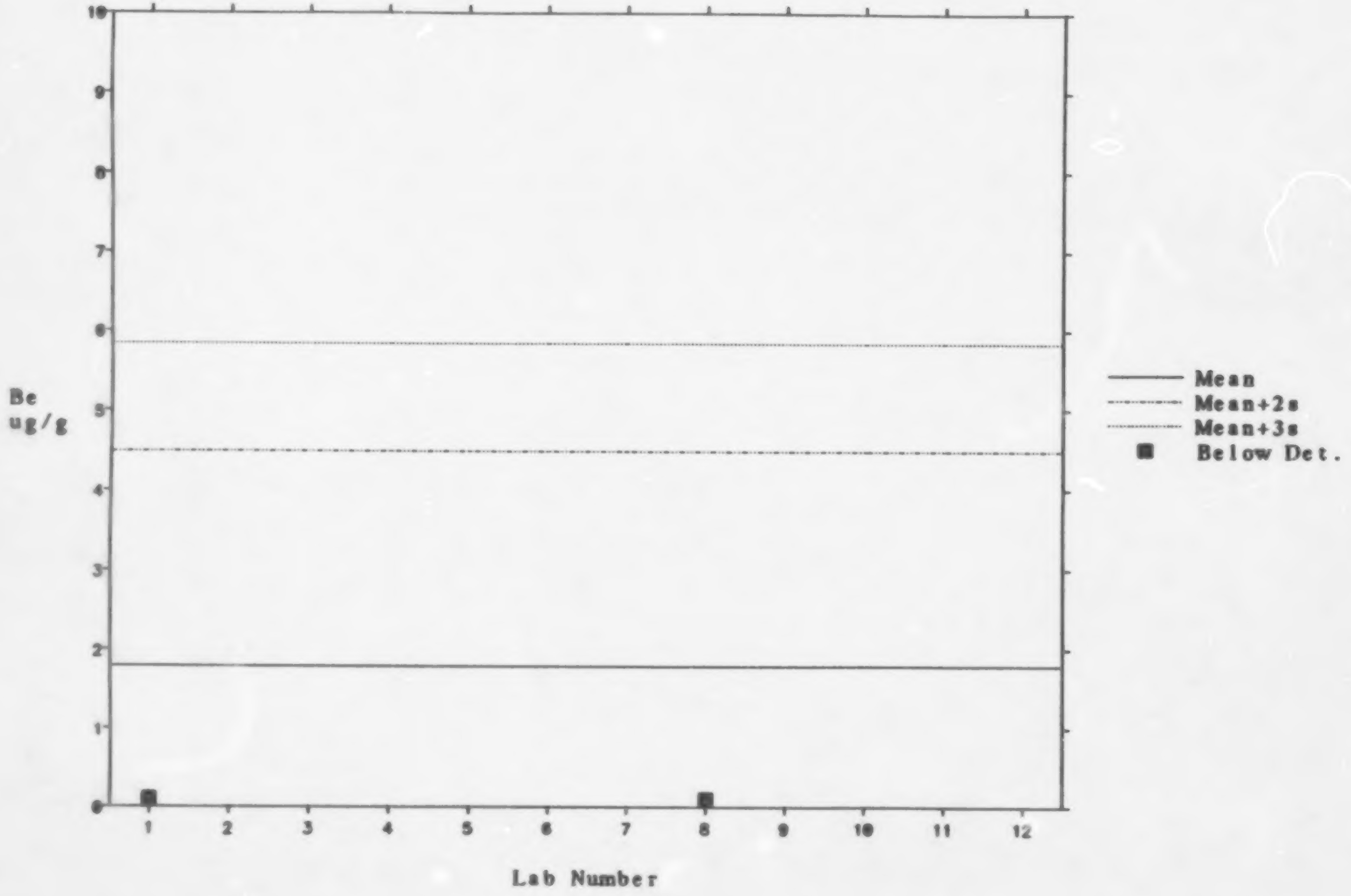


Figure 49. Analysis of Be in Sludge F



Figures 44 to 49 indicate that all the reported Be data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods. The Be detection limits for the participating labs varied widely by more than one order of magnitude.

The Be data shows good agreement between some of the labs and for the different methodologies used. The low number of samples analyzed and reporting labs limits the ability to further interpret this data.

4.1.1.6 Cd

Seven labs reported Cd in the ICP standard (Figure 50), all but two labs were within the control limits. Nine labs reported Cd for the digestate 3 and eight labs for the digestate 4 samples (Figures 51 and 52). Digestate 3 and 4 data (Figures 51 and 52) were all within the control limits, indicating very good agreement between the participating labs. Seven labs reported Cd for the standard A and eight labs for the standard B samples (Figures 53 and 54). Figure 53 shows that all labs reported data within Standards A's warning limit, with one lab reporting data (using other methods) between the control and warning limits. Standard B values (Figure 54) show that all but one lab had data within the warning limits, with 6 labs reporting some data both inside and outside the control limits. Figure 54 also shows that the results of the ARC-CPA method were more frequently located within the warning limits than the results of other methods.

The statistical data for Cd in sludges A to F are shown in Table 7, and their control charts in Figures 55 to 60. Table 7 shows that results (above detection) were reported for all sludges, with number of analyses varying from 7 to 26 samples. The mean Cd values ranged from 3.9 mg/kg for sludge D to 7.2 for sludges B and C. The coefficient of variation of the sludges varied from 59 to 208% and it did not seem to be related to the number of reported results or the measured concentrations.

Figures 55 to 60 indicate that all the reported Cd data fell within the sludges' control limits, with some apparent differences between the ARC-CPA and other methods.

Figure 50. Mean and Confidence Intervals for Cd in an ICP Standard

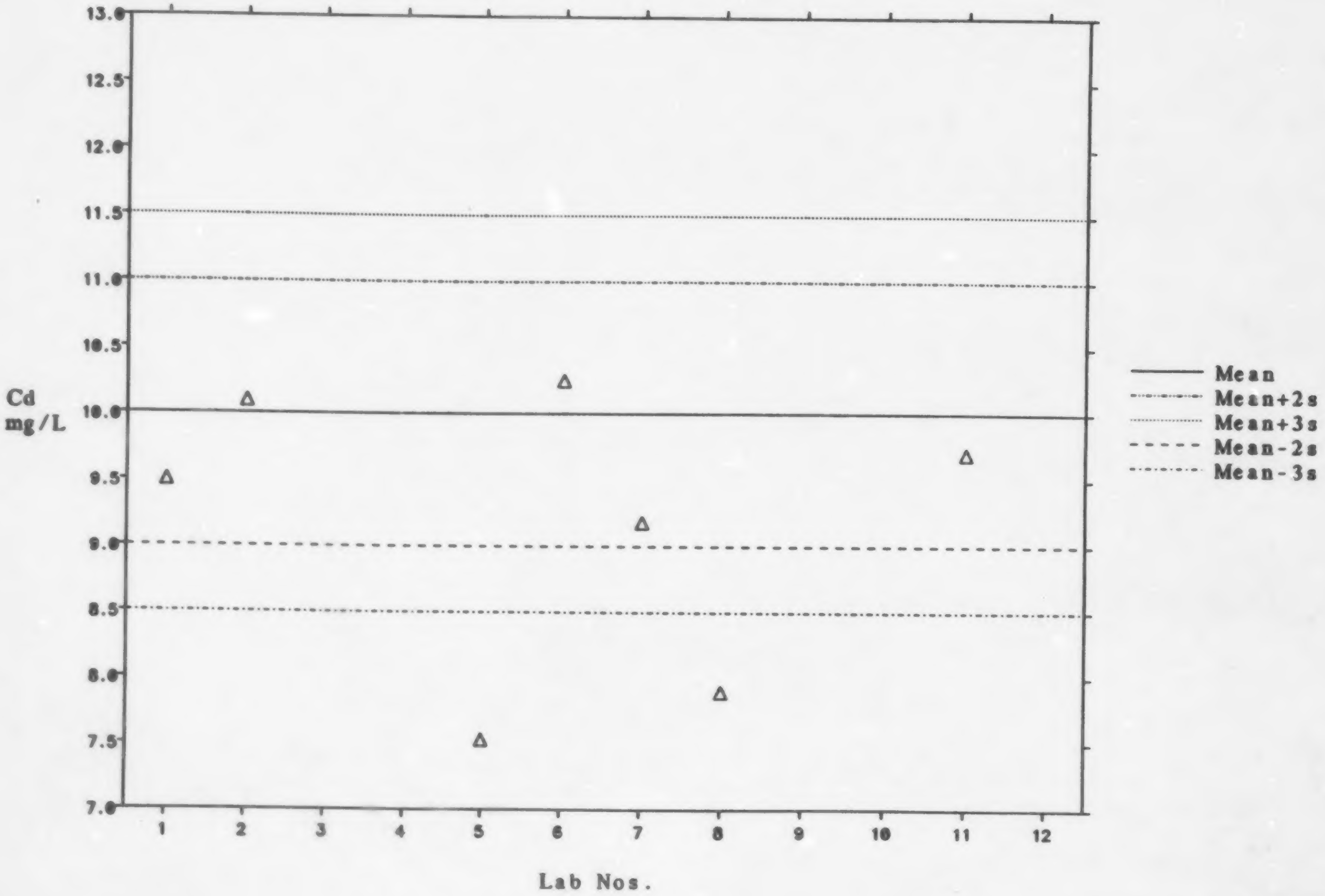


Figure 51. Mean and Confidence Intervals for Cd in Digestate 3

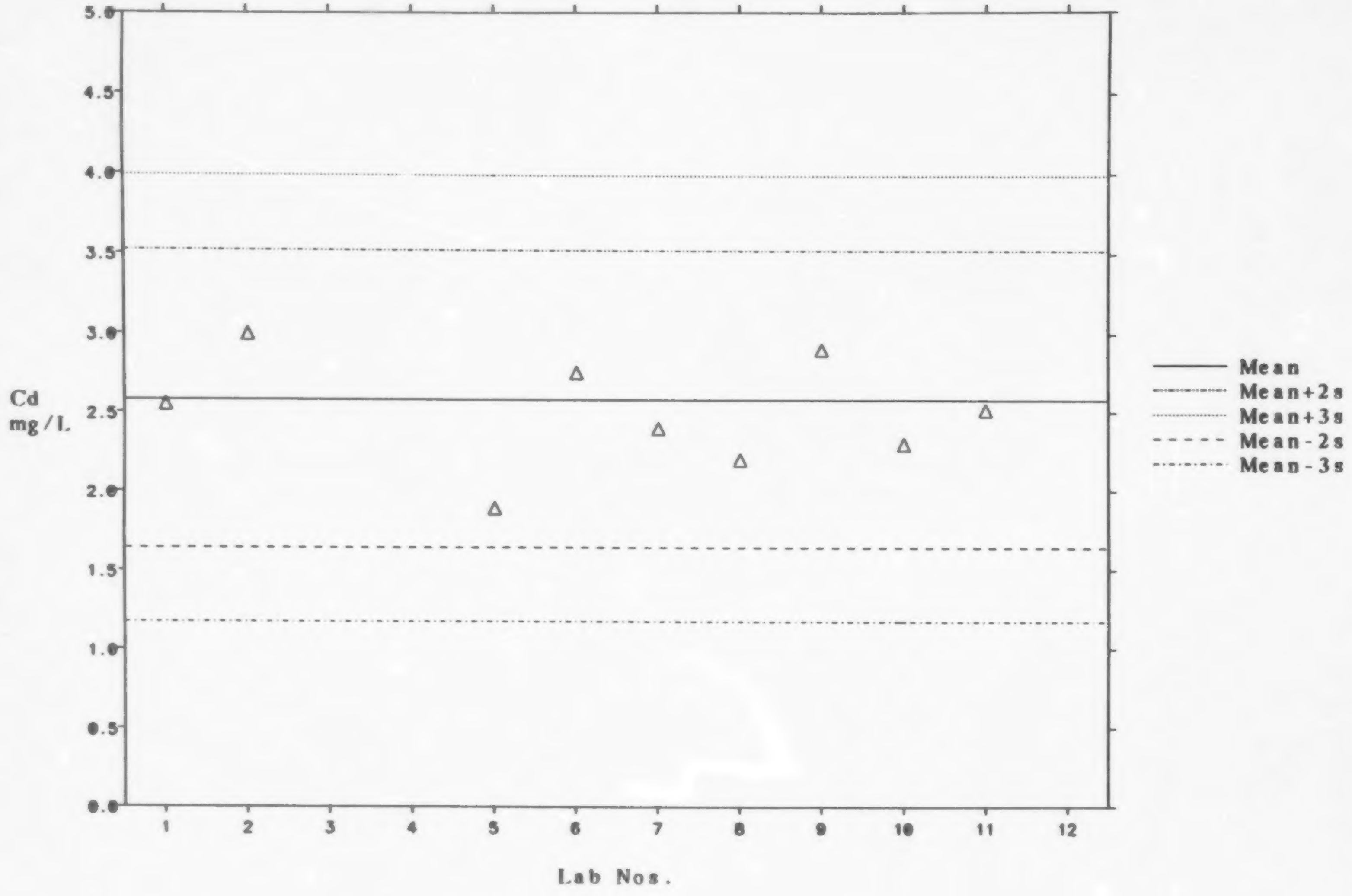


Figure 52. Mean and Confidence Intervals for Cd in Digestate 4

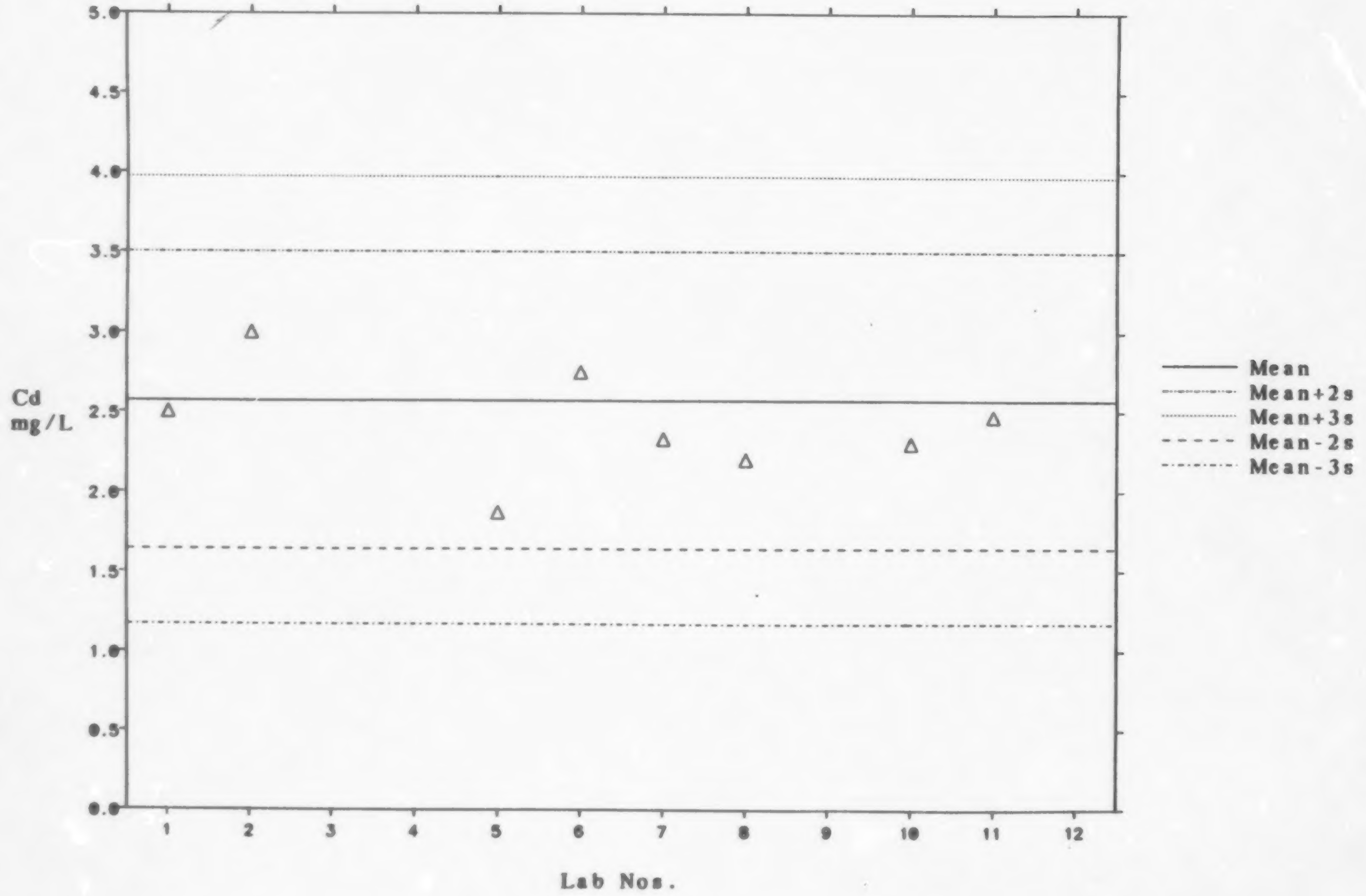


Figure 53. Mean and Confidence Intervals for Cd in Standard A

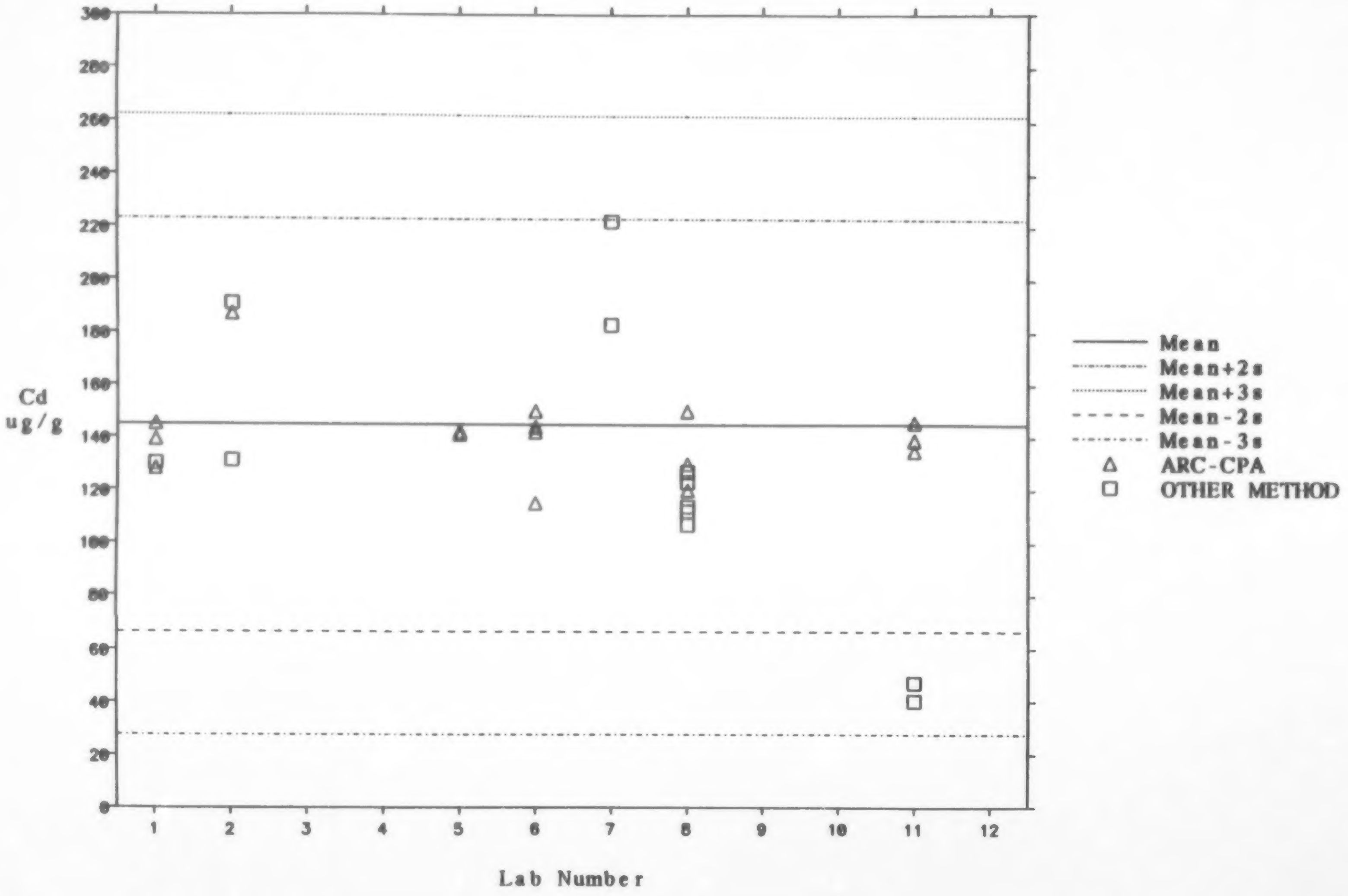


Figure 54. Mean and Confidence Intervals for Cd in Standard B

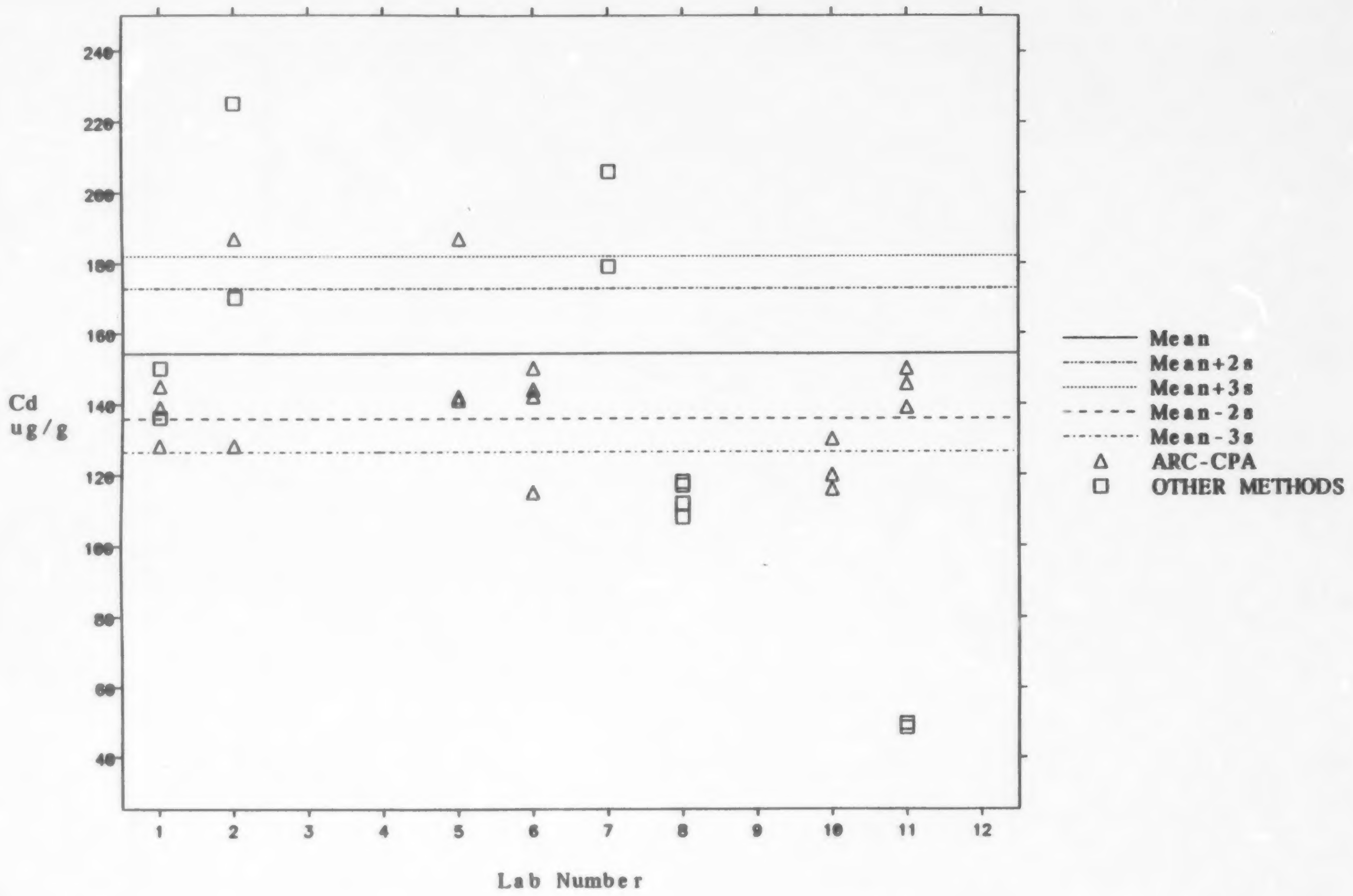


Table 7. Statistical Parameters for Cd (mg/Kg) in Sludges A-F

Statistic	Sludge					
	A	B	C	D	E	F
Count	13.00	23.00	20.00	28.00	7.00	26.00
Sum	60.50	165.00	145.00	110.00	30.00	157.00
Mean	4.66	7.18	7.23	3.93	4.28	6.03
SEM	1.74	0.89	2.56	1.54	2.49	1.83
Median	2.00	6.50	1.15	2.00	2.19	2.00
Variance	39.30	18.00	131.00	66.50	43.60	86.90
StDev	6.27	4.24	11.50	8.16	6.60	9.32
Maximum	18.80	16.70	35.70	43.60	19.00	35.70
Minimum	0.17	2.00	0.40	0.05	0.36	0.40
Range	18.60	14.70	35.30	43.60	18.60	35.30
Skewness	1.57	0.86	1.96	4.59	2.47	2.36
Kurtosis	1.05	0.06	2.49	22.60	6.26	5.12
CV(%)	135.00	59.10	158.00	208.00	154.00	155.00

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 55. Analysis of Cd in Sludge A

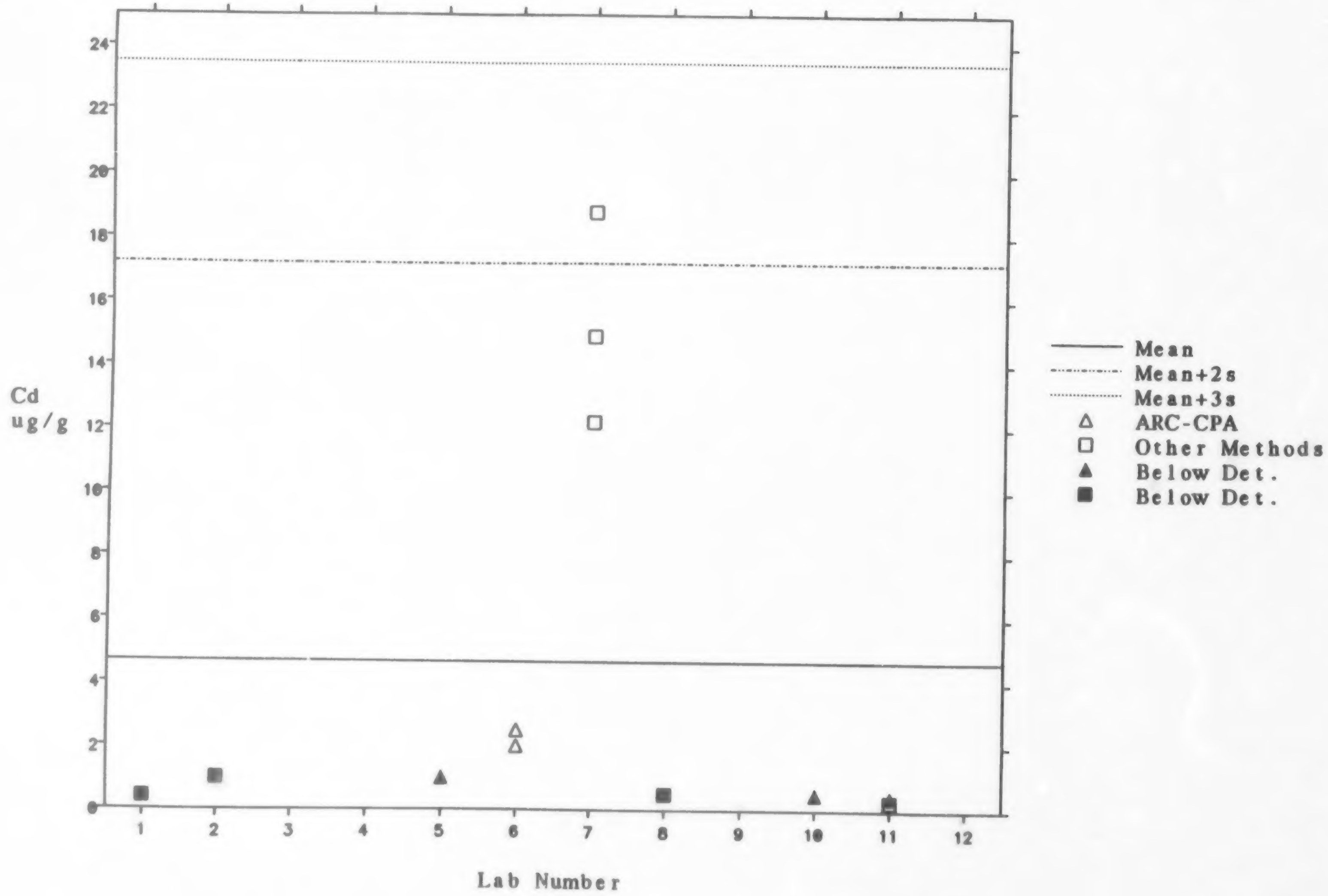


Figure 56. Analysis of Cd in Sludge B

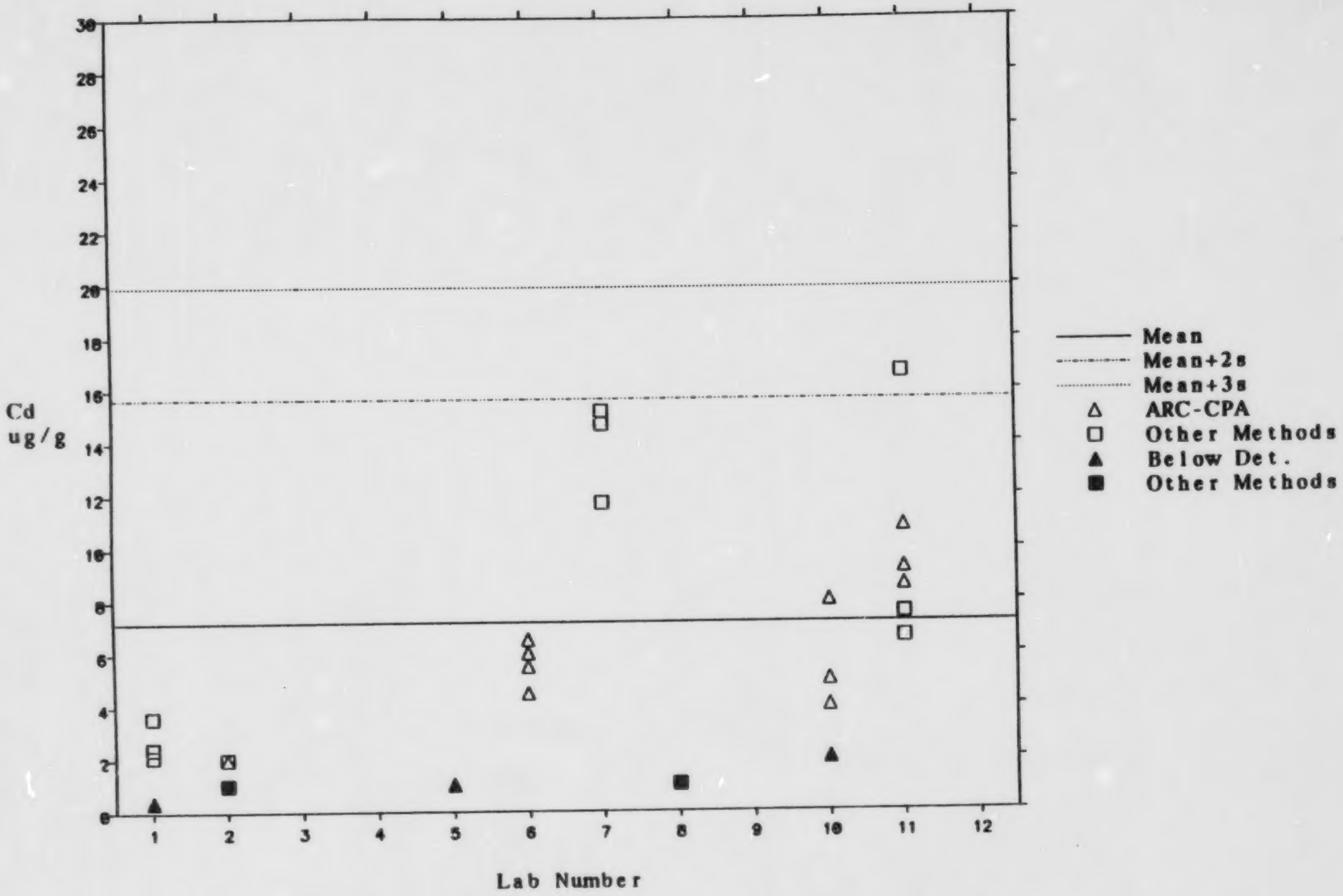


Figure 57. Analysis of Cd in Sludge C

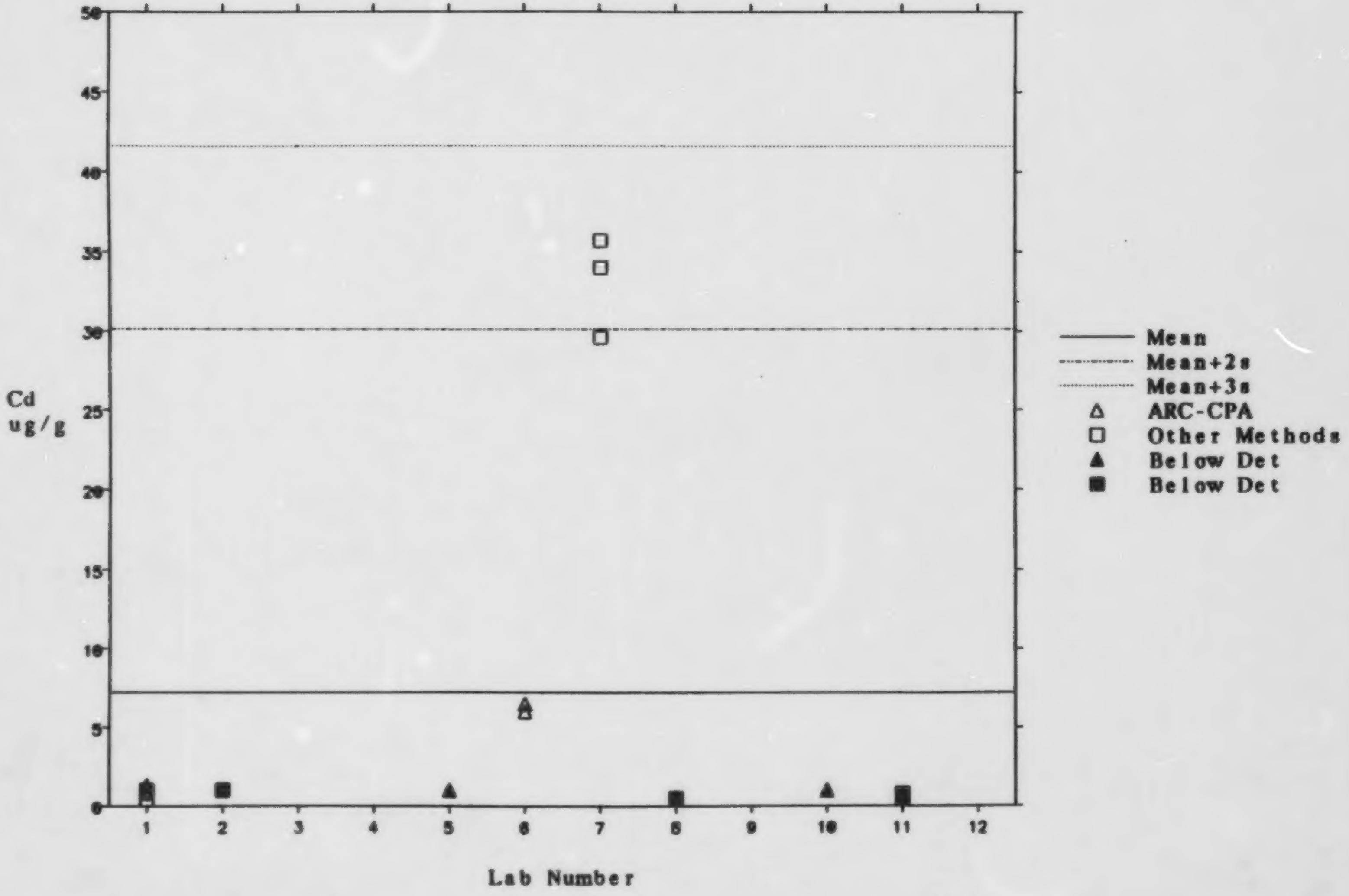


Figure 58. Analysis of Cd in Sludge D

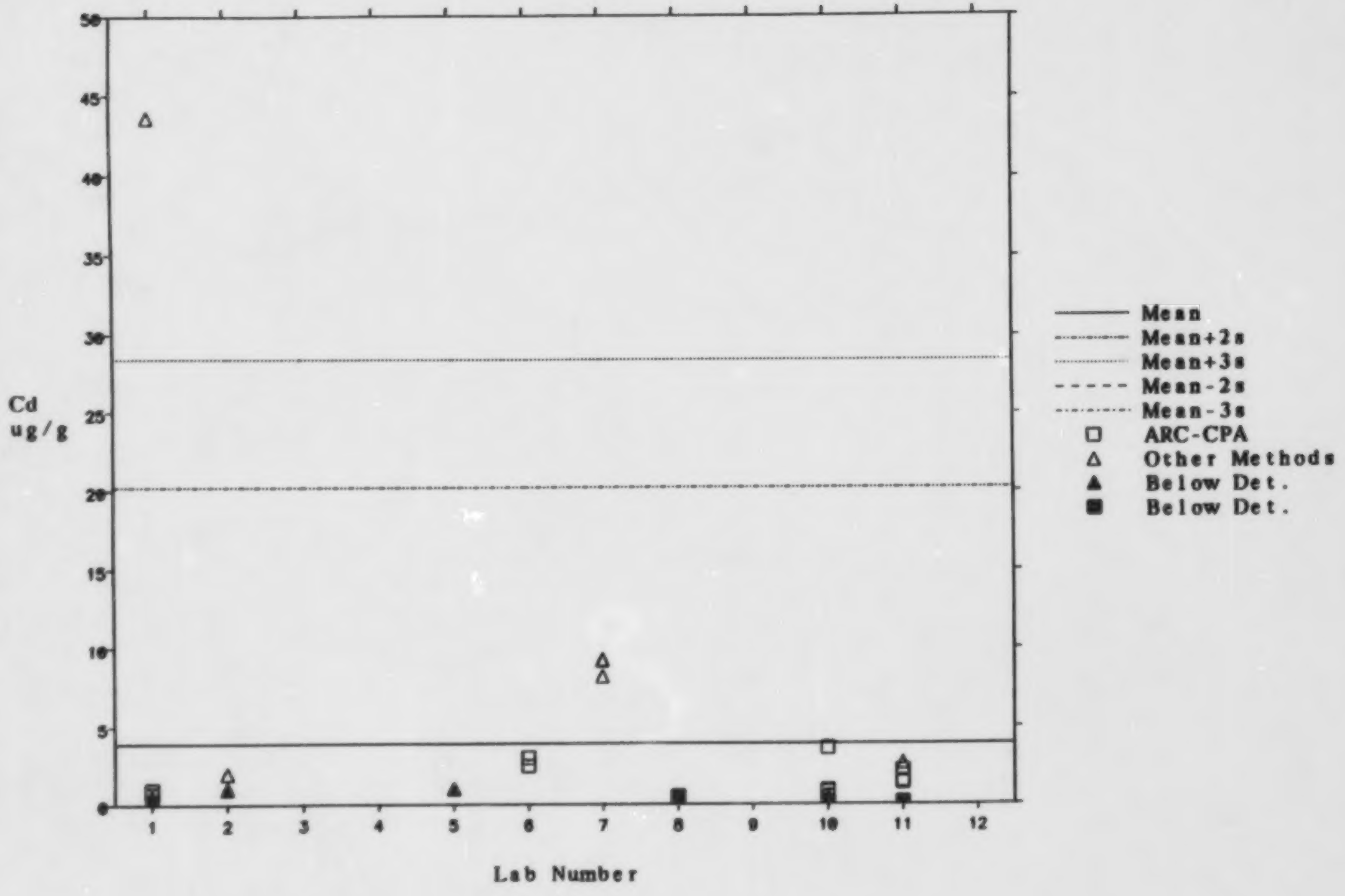


Figure 59. Analysis of Cd in Sludge E

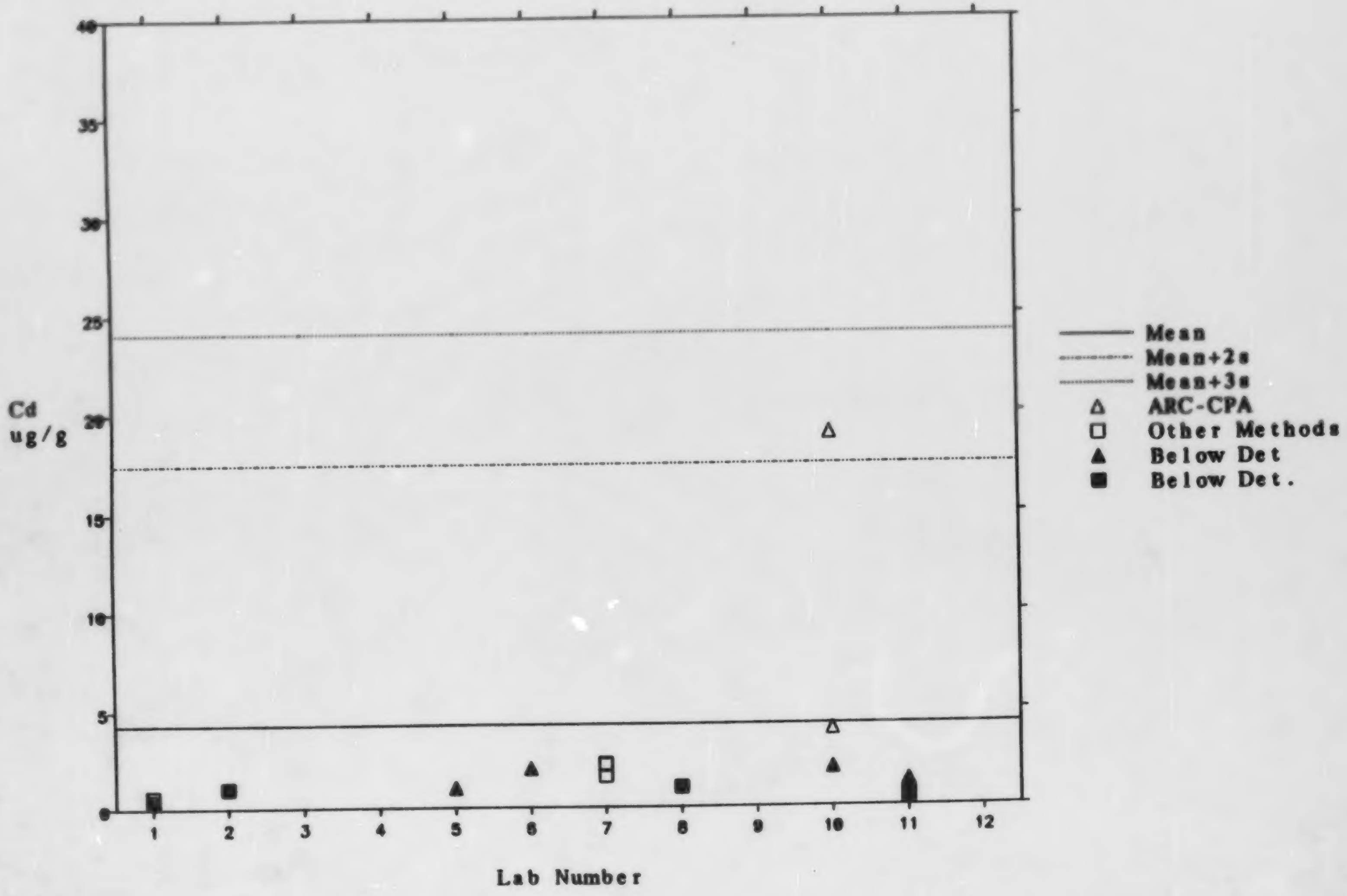
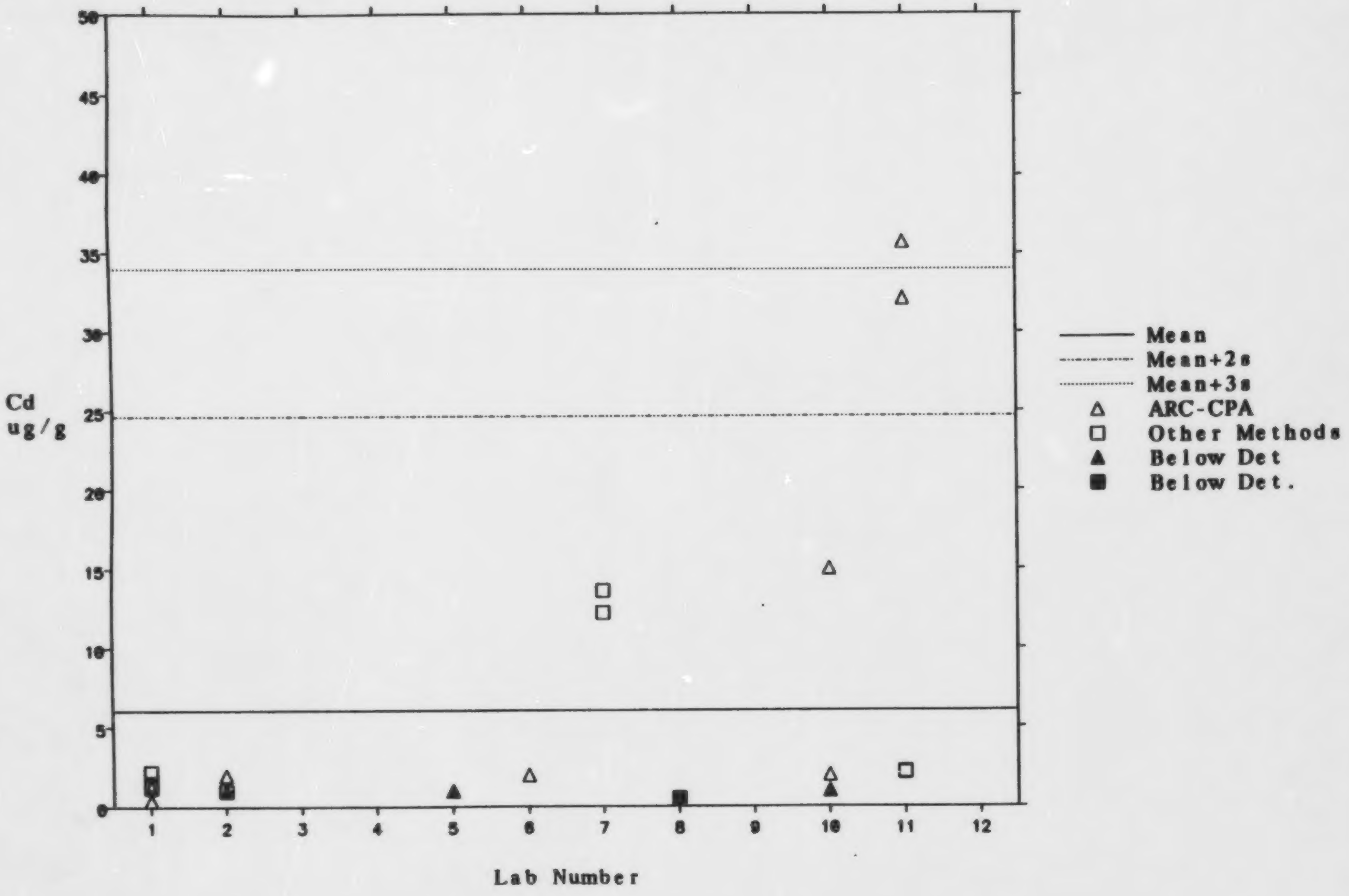


Figure 60. Analysis of Cd in Sludge F



The Cd data shows some agreement between the different labs and some differences between the different methodologies used. The variability did not seem to be dependent on the number of analyses performed or the Cd concentration.

4.1.1.7 Co

Seven labs reported Co in the ICP standard (Figure 61), all but two labs were within the control limits. Nine labs reported Co for the digestate samples (Figures 62 and 63). Digestate 3 and 4 data (Figures 62 and 63) were all within the control limits except for one lab whose data fell between the control and warning limits. Eight labs reported Co for the standard A and B samples (Figures 64 and 65). Figure 64 shows that all but one lab reported data within Standards A's warning limit, with six labs reporting data within and outside these limits. Standard B values (Figure 65) show that all but two labs had their data outside the warning limits, with the 2 labs reporting some data both inside and outside the control limits. Figure 65 indicates the possibility of having a higher mean Co value for Standard B than reported. These results also show that the results of the ARC-CPA method were generally similar to those from other methods.

The statistical data for Co in sludges A to F are shown in Table 8, and their control charts in Figures 66 to 71. Table 8 shows that results (above detection) were reported for all sludges, with number of analyses varying from 6 to 44 samples. The mean Co values ranged from 2.8 mg/kg for sludge E to 62.9 for sludge B. The coefficient of variation of the sludges varied from 14 to 125% and it seems to be related to the number of reported results and the measured concentrations.

Figures 66 to 77 indicate that all the reported Co data fell within the sludges' control limits except for lab 7 and sludges A and B, with no differences between the ARC-CPA and other methods.

The Co data shows good agreement between the different labs and for the different methodologies used.

Figure 61. Mean and Confidence Intervals for Co in an ICP Standard

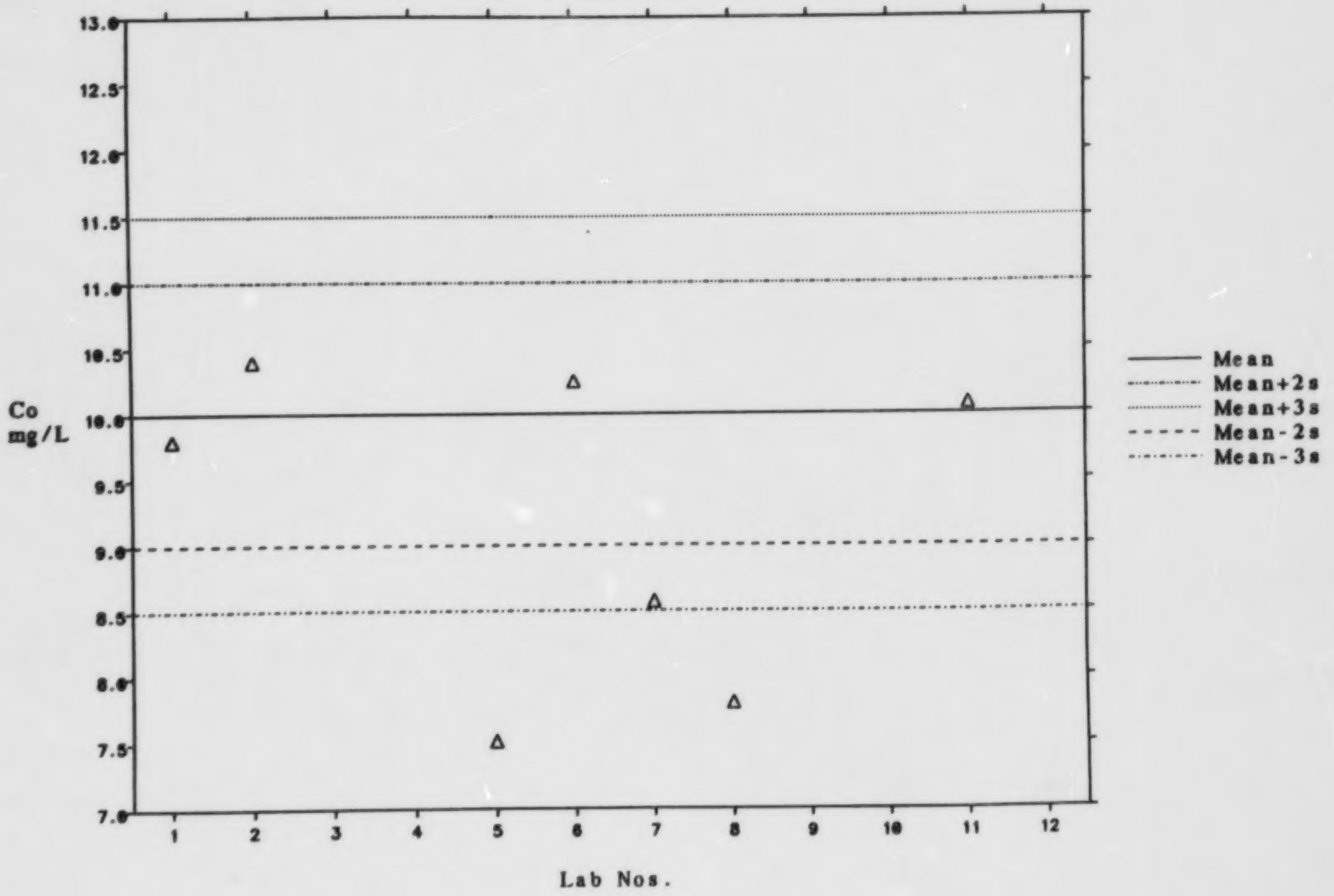


Figure 62. Mean and Confidence Intervals for Co in Digestate 3

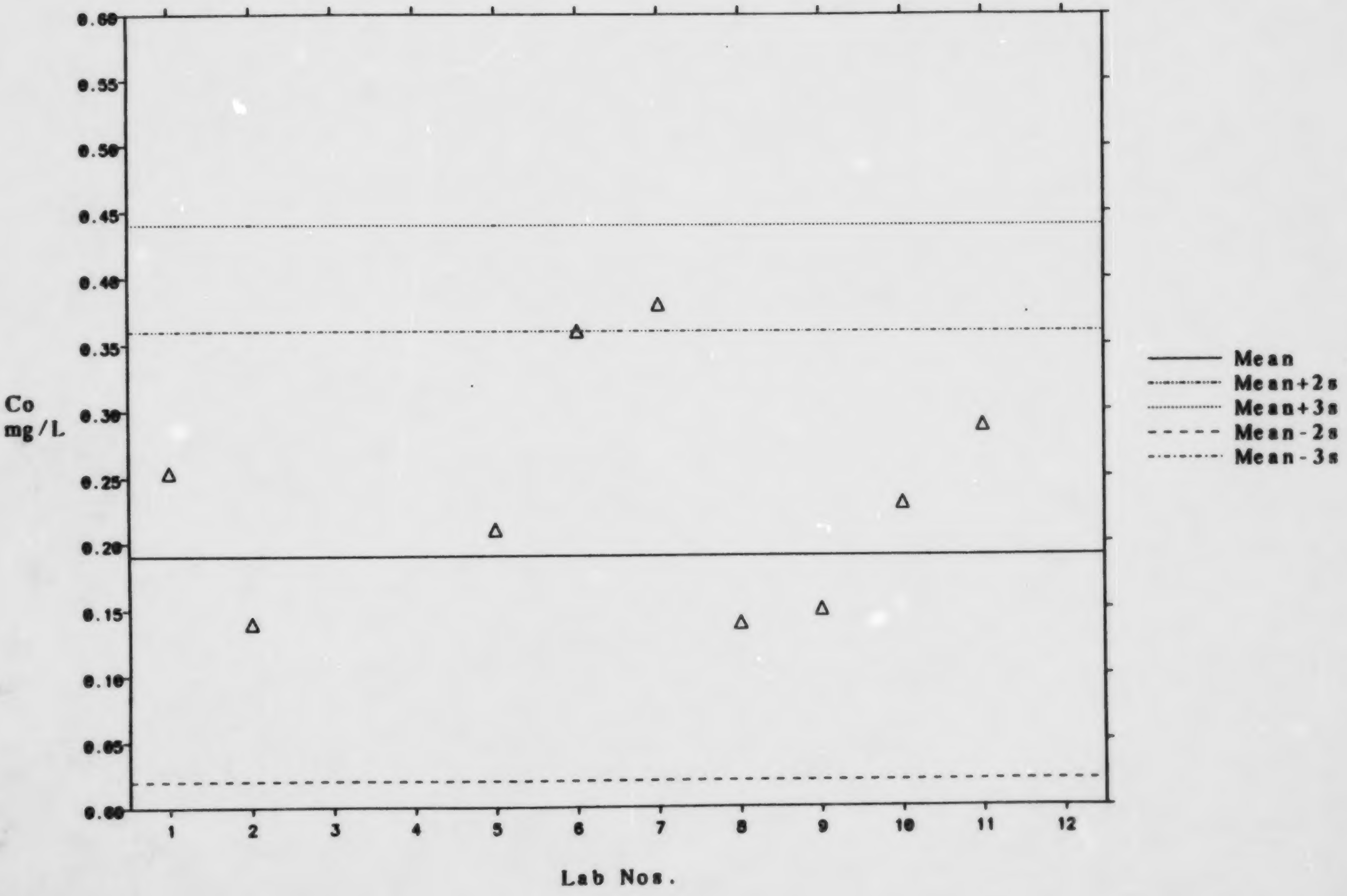


Figure 63. Mean and Confidence Intervals for Co in Digestate 4

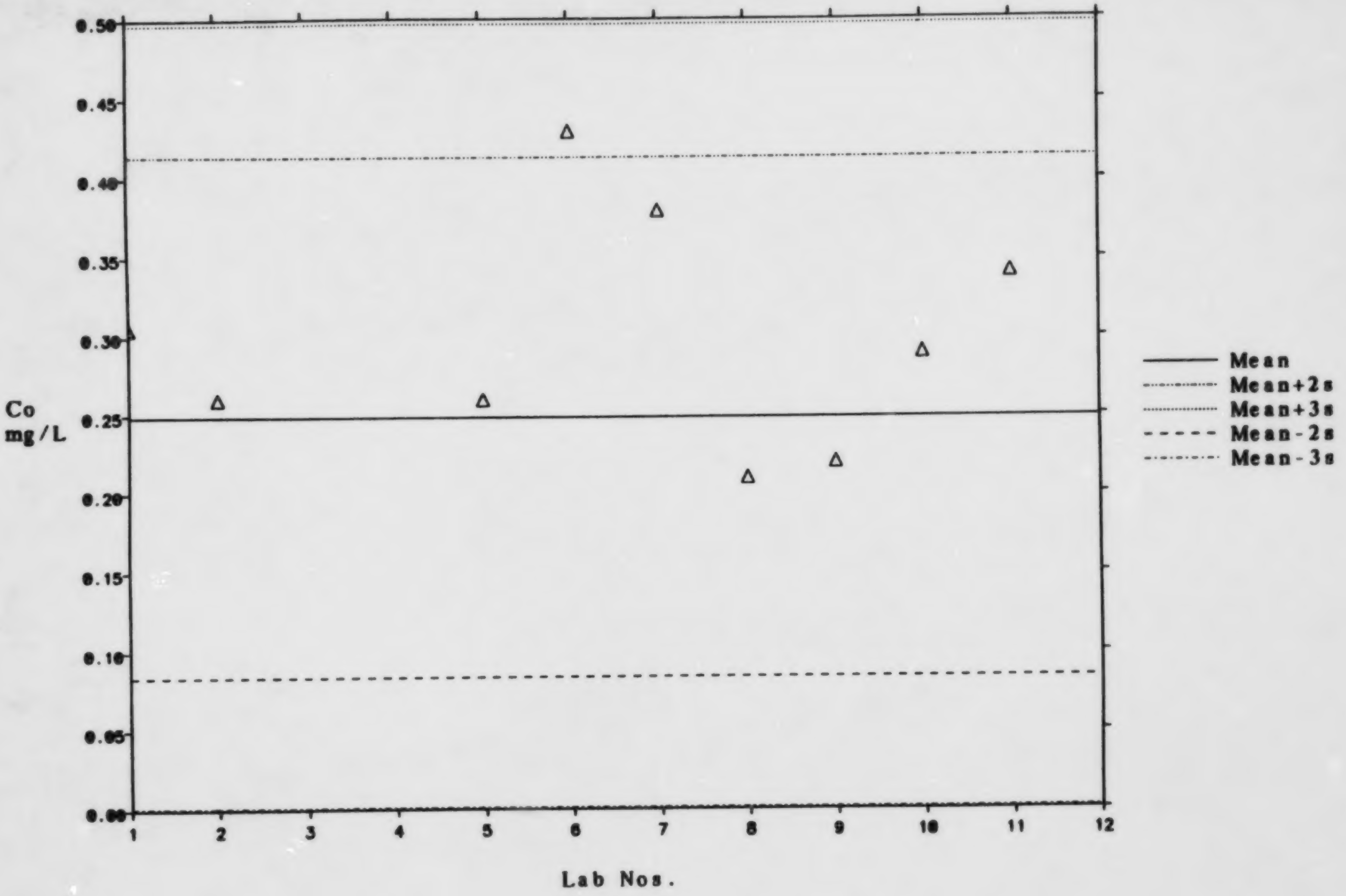


Figure 64. Mean and Confidence Intervals for Co in Standard A

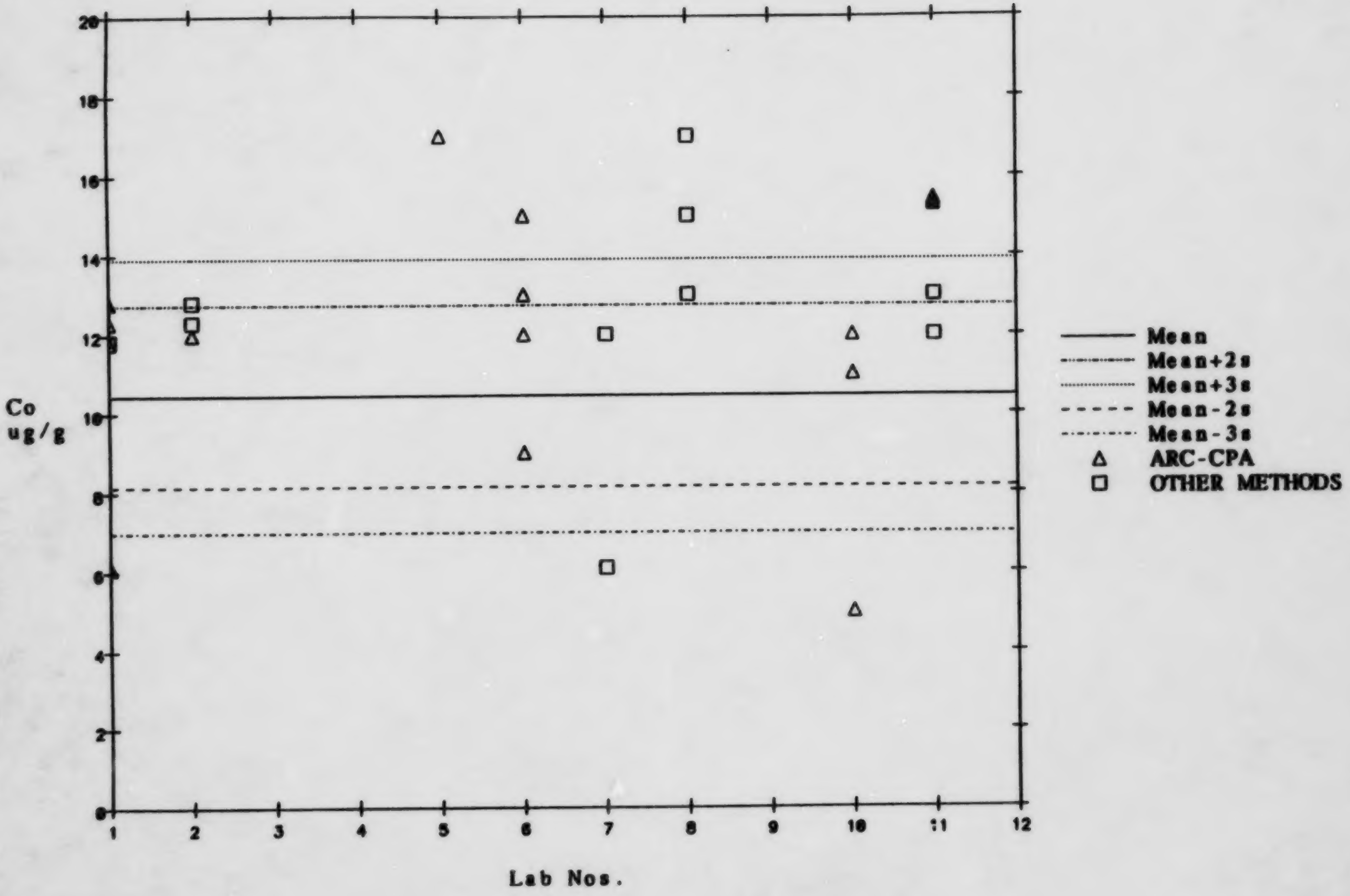


Figure 65. Mean and Confidence Intervals for Co in Standard B

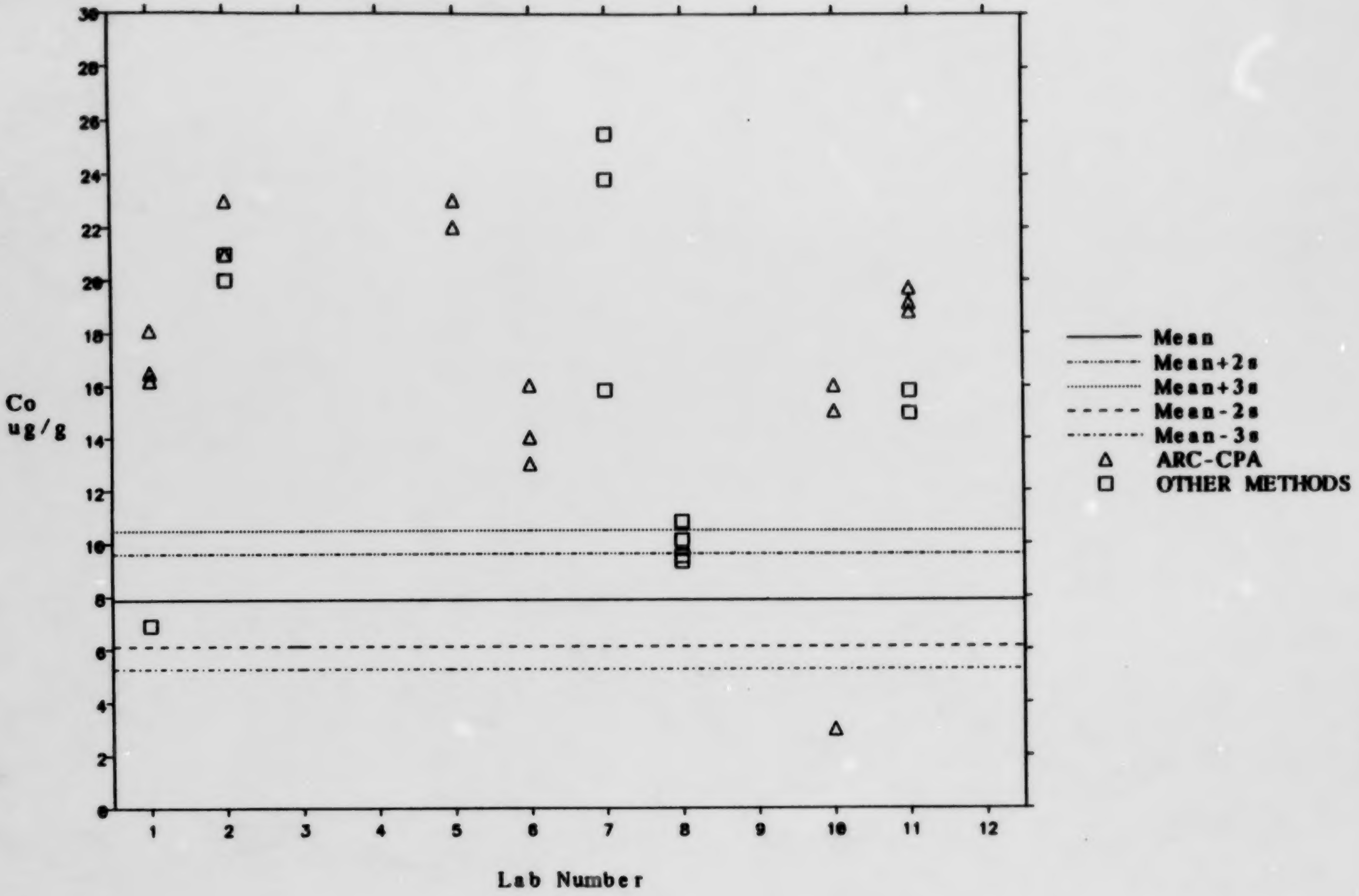


Table 8. Statistical Parameters for Co (mg/Kg) in Sludges A-F

Statistic	A	B	C	D	E	F
Count	29.00	44.0	30.00	44.00	6.000	33.00
Sum	152.00	2770.0	260.00	878.00	16.700	317.00
Mean	5.26	62.9	8.68	20.00	2.780	9.62
SEM	1.15	1.4	1.98	0.85	0.759	1.08
Median	3.00	61.5	3.00	20.00	3.210	8.00
Variance	38.50	79.8	118.00	31.90	3.450	38.70
StDev	6.21	8.9	10.90	5.65	1.860	6.22
Maximum	31.40	90.7	37.80	35.00	4.460	30.40
Minimum	1.00	47.0	0.94	12.80	0.600	2.00
Range	30.40	43.7	36.90	22.20	3.860	28.40
Skewness	3.09	0.8	1.71	0.78	-0.252	2.25
Kurtosis	1.30	0.9	2.00	-0.03	-2.740	5.20
CV	118.00	14.2	125.00	28.30	66.900	64.70

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 66. Analysis of Co in Sludge A

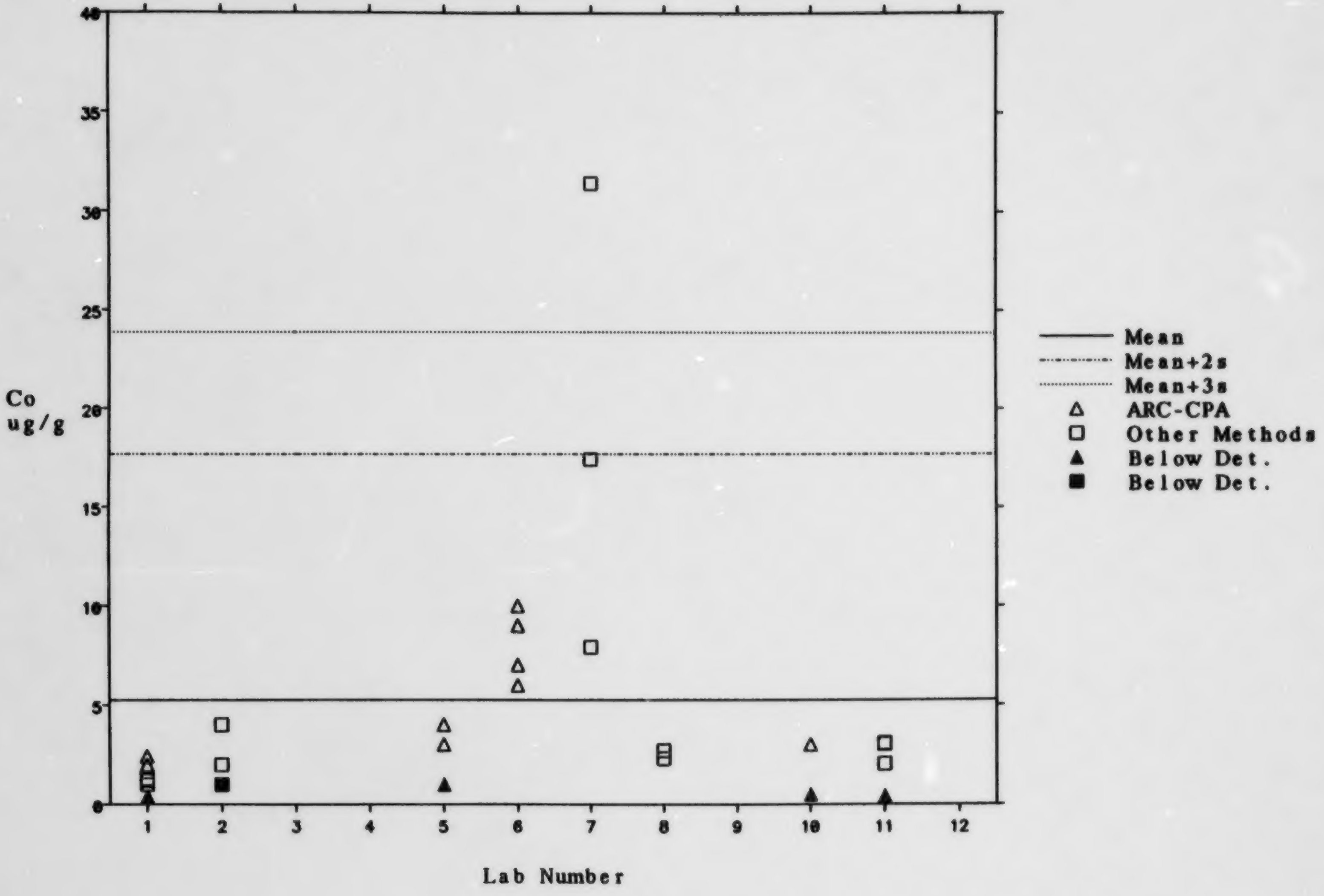


Figure 67. Analysis of Co in Sludge B

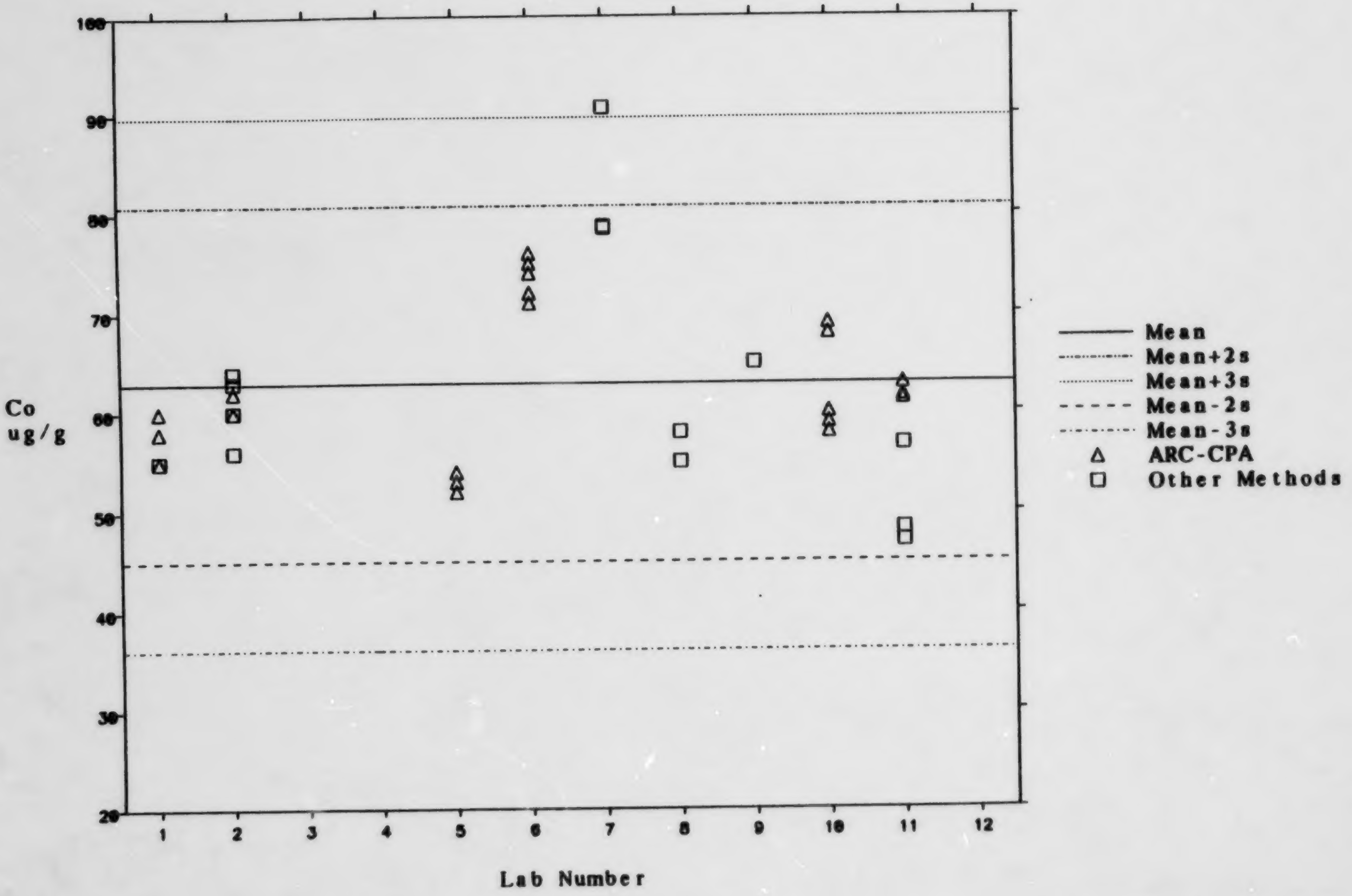
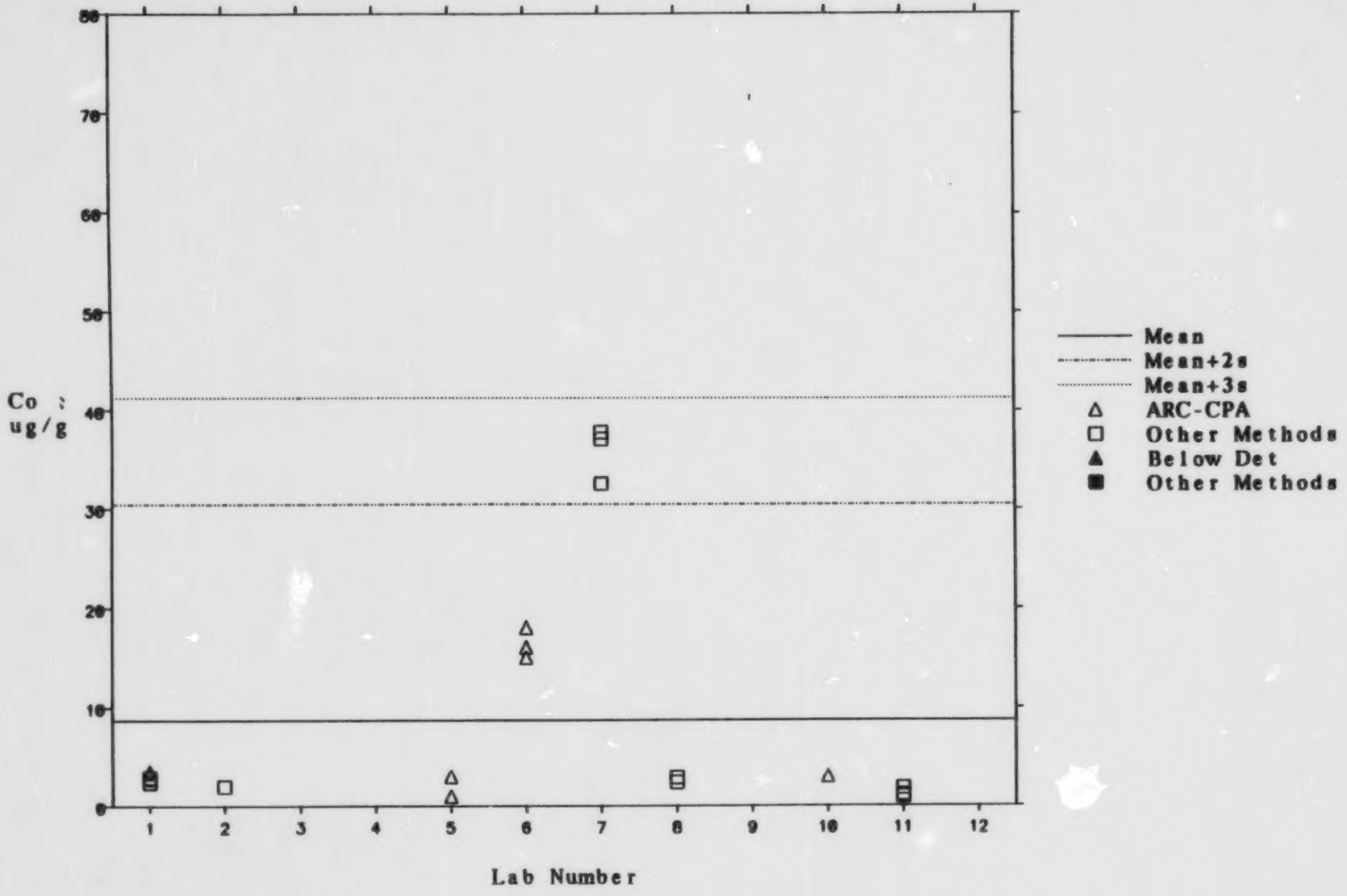


Figure 68. Analysis of Co in Sludge C



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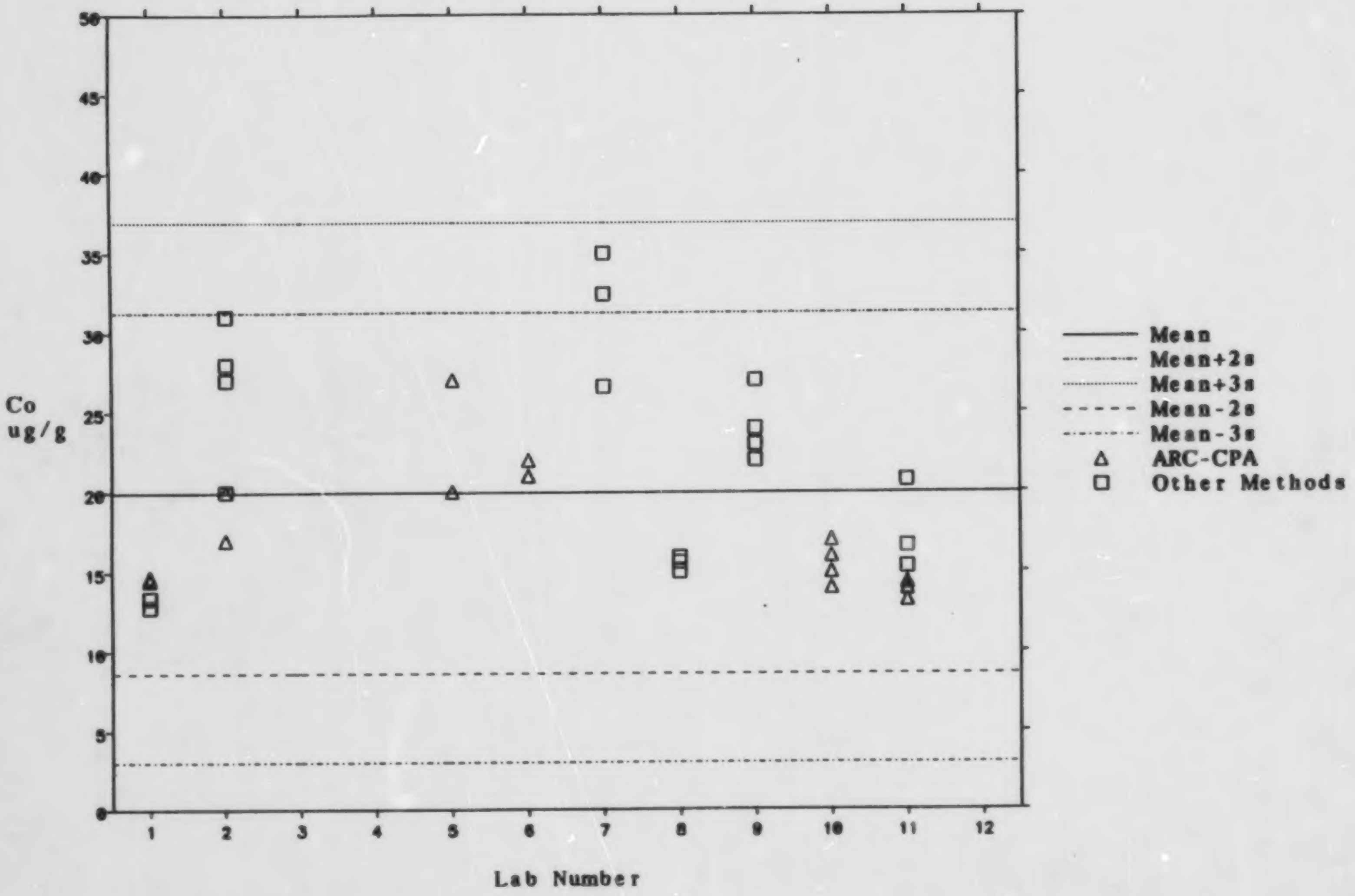


Figure 69. Analysis of Co in Sludge D

Figure 70. Analysis of Co in Sludge E

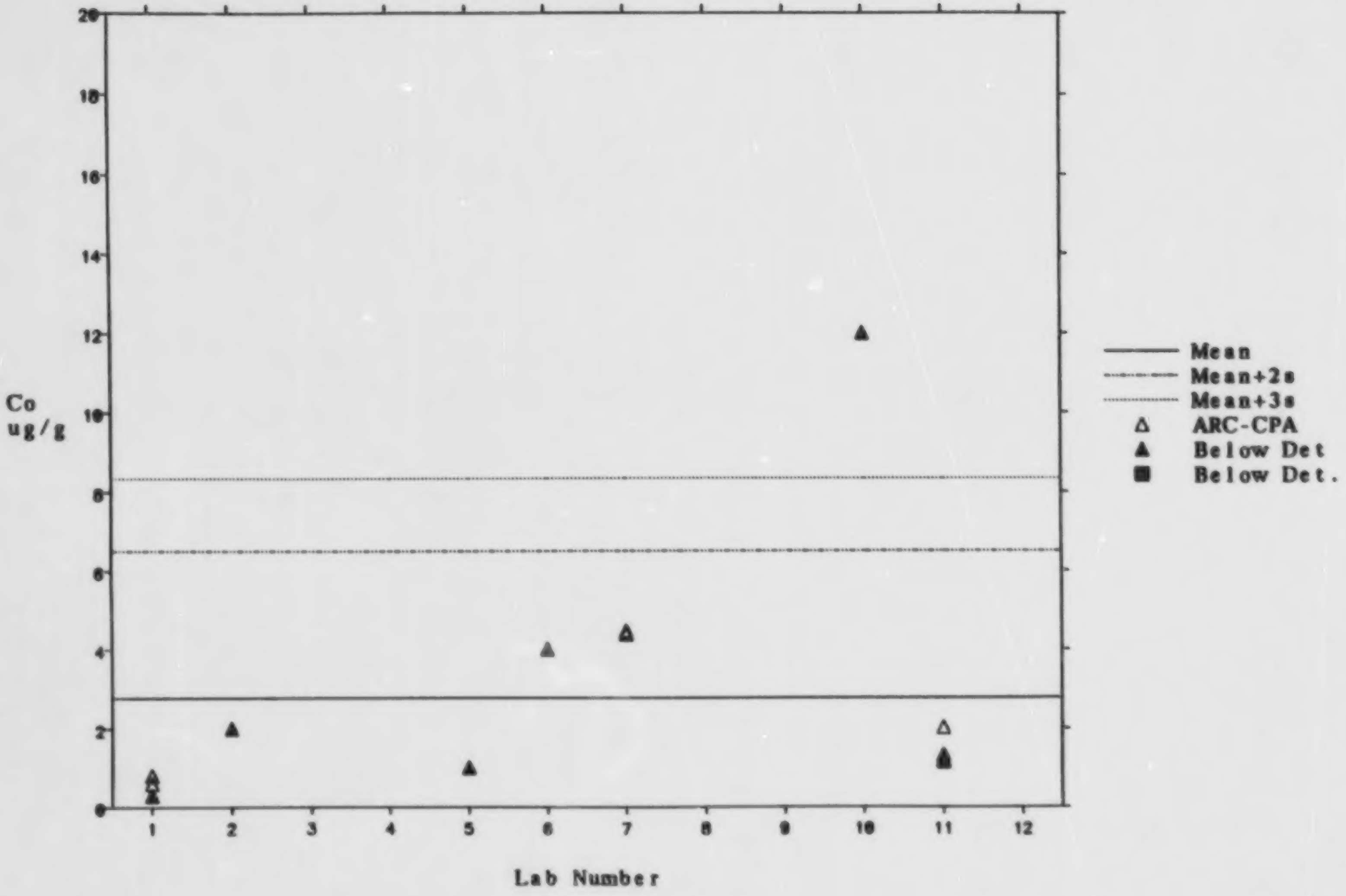
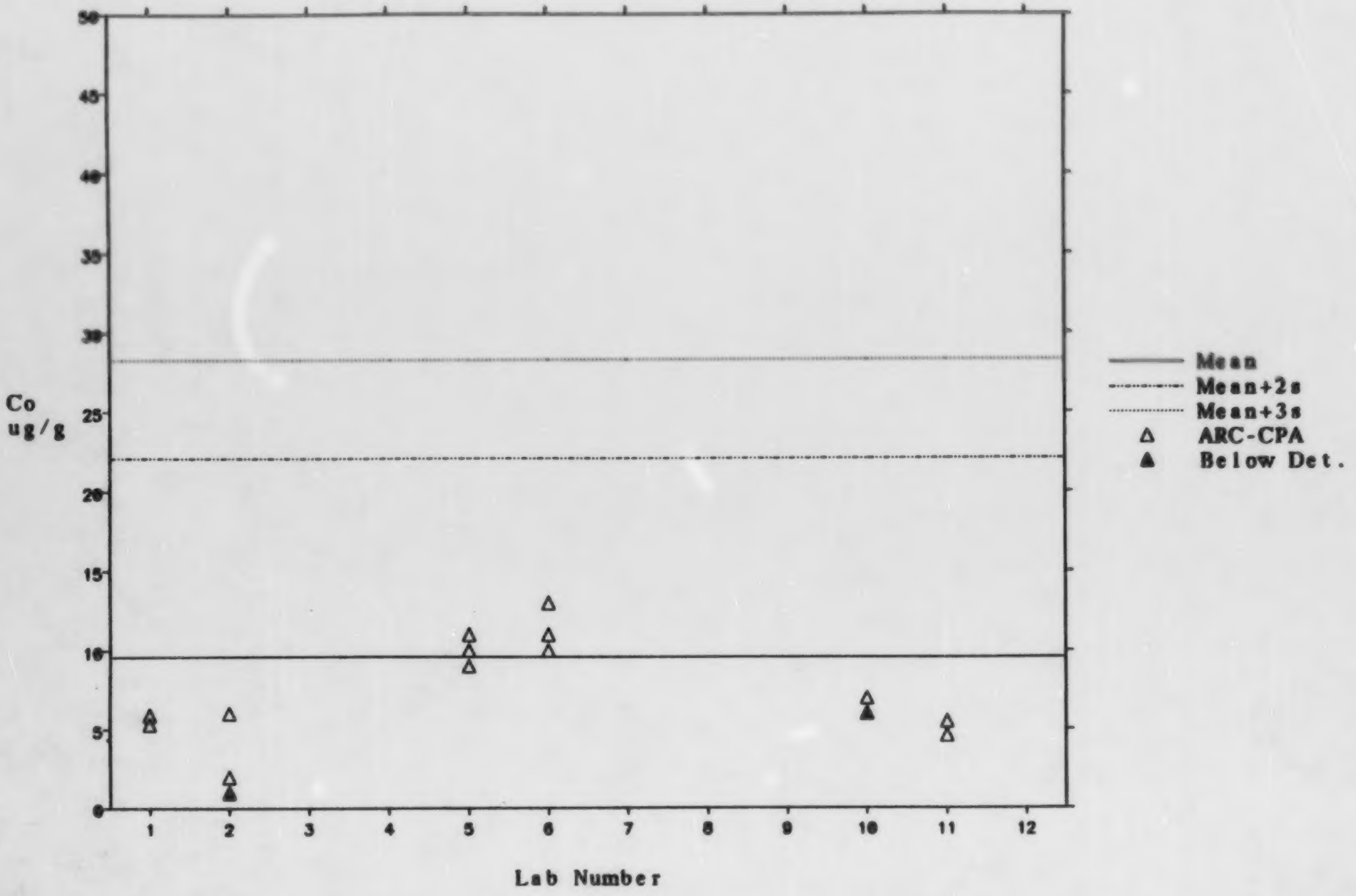


Figure 71. Analysis of Co in Sludge F



4.1.1.8 Cr

Eight labs reported Cr in the ICP standard (Figure 72), all but three labs were within the control limits. One of the three outlier labs was between the control and warning limit, while the other two were lower than the lowest warning limit. Nine labs reported Cr for the digestate samples (Figures 73 and 74). Digestate 3 and 4 data (Figures 73 and 74) were all within the control limits, evidence of very good agreement between all the labs. Eight labs reported Cr for the standard A and B samples (Figures 75 and 76). Figure 75 shows that all labs reported data within Standards A's warning limits and only 2 labs with data outside and inside those limits. Standard B values (Figure 76) show that 3 labs were completely outside the warning limits, 4 labs with data inside and outside the limits, and only 1 lab completely within these limits. Standard A data shows much more agreement between the labs and the actual value than data from Standard B. Figures 75 and 76 also show that the results of the ARC-CPA method were similar to those of other methods.

The statistical data for Cr in sludges A to F are shown in Table 9, and their control charts in Figures 77 to 82. Table 9 shows that results (above detection) were reported for all sludges, with number of analyses varying from 34 to 42 samples. The mean Cr values ranged from 39.1 mg/kg for sludge F to 277 for sludge B. The coefficient of variation of the sludges varied from 14.9 to 49.5% and it seems to be inversely related to the number of reported results and the measured concentrations. These CV values are quite acceptable for such a diverse and non homogeneous material.

Figures 77 to 82 indicate that all the reported Cr data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Cr data shows good agreement between the different labs and for the different methodologies used. The variability seems to be

Figure 72. Mean and Confidence Intervals for Cr in an ICP Standard

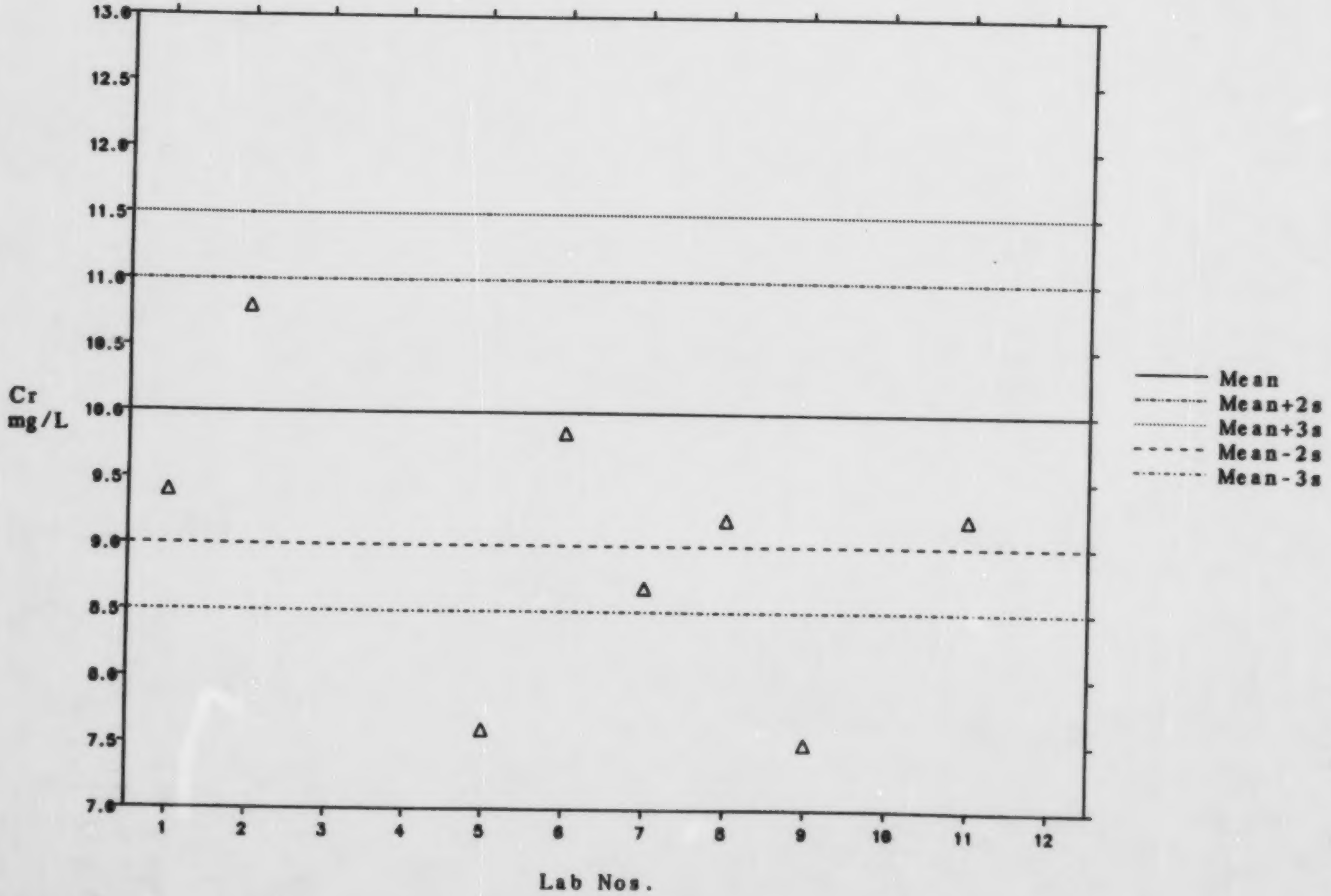


Figure 73. Mean and Confidence Intervals for Cr in Digestate 3

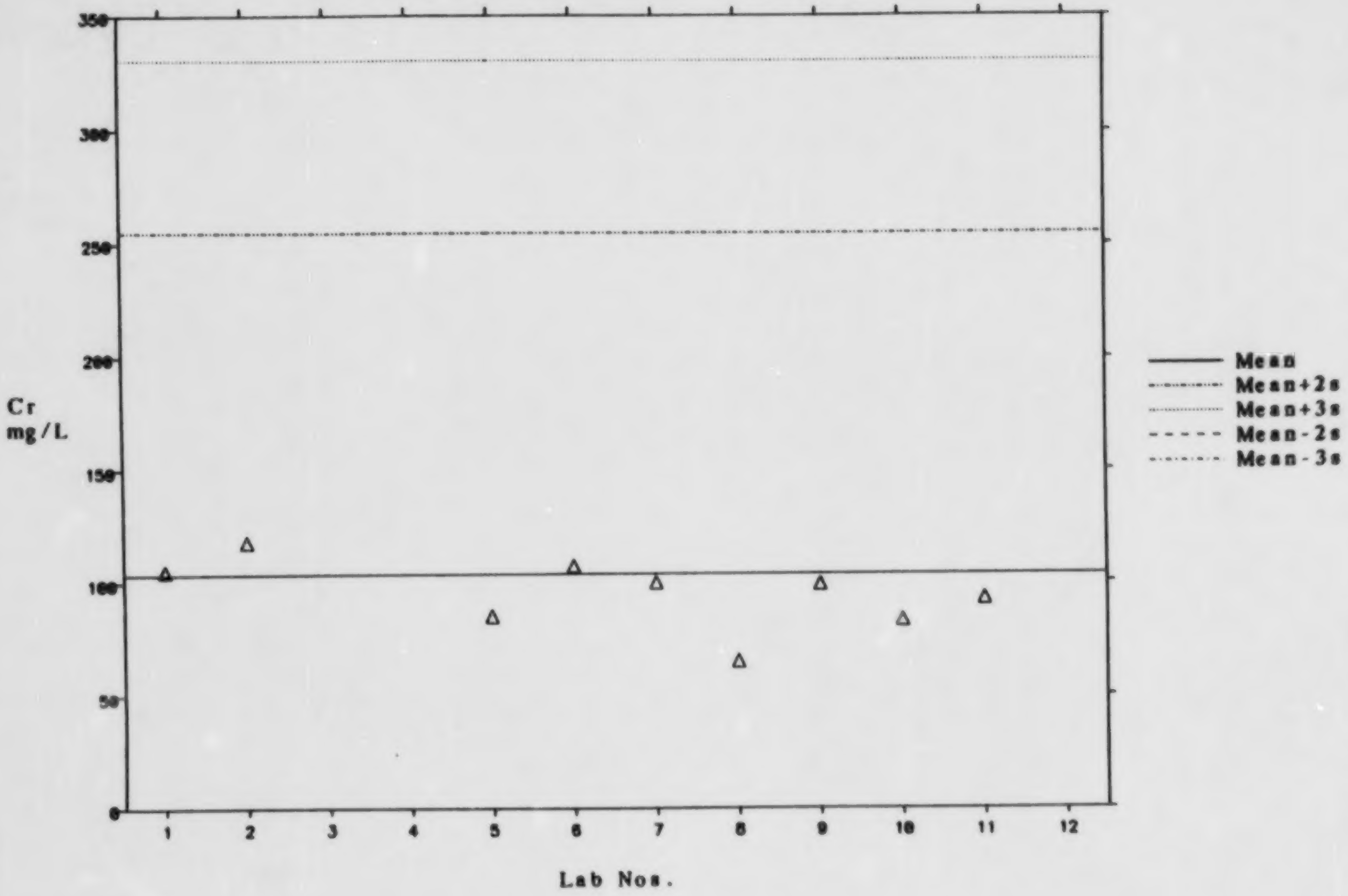


Figure 74. Mean and Confidence Intervals for Cr in Digestate 4

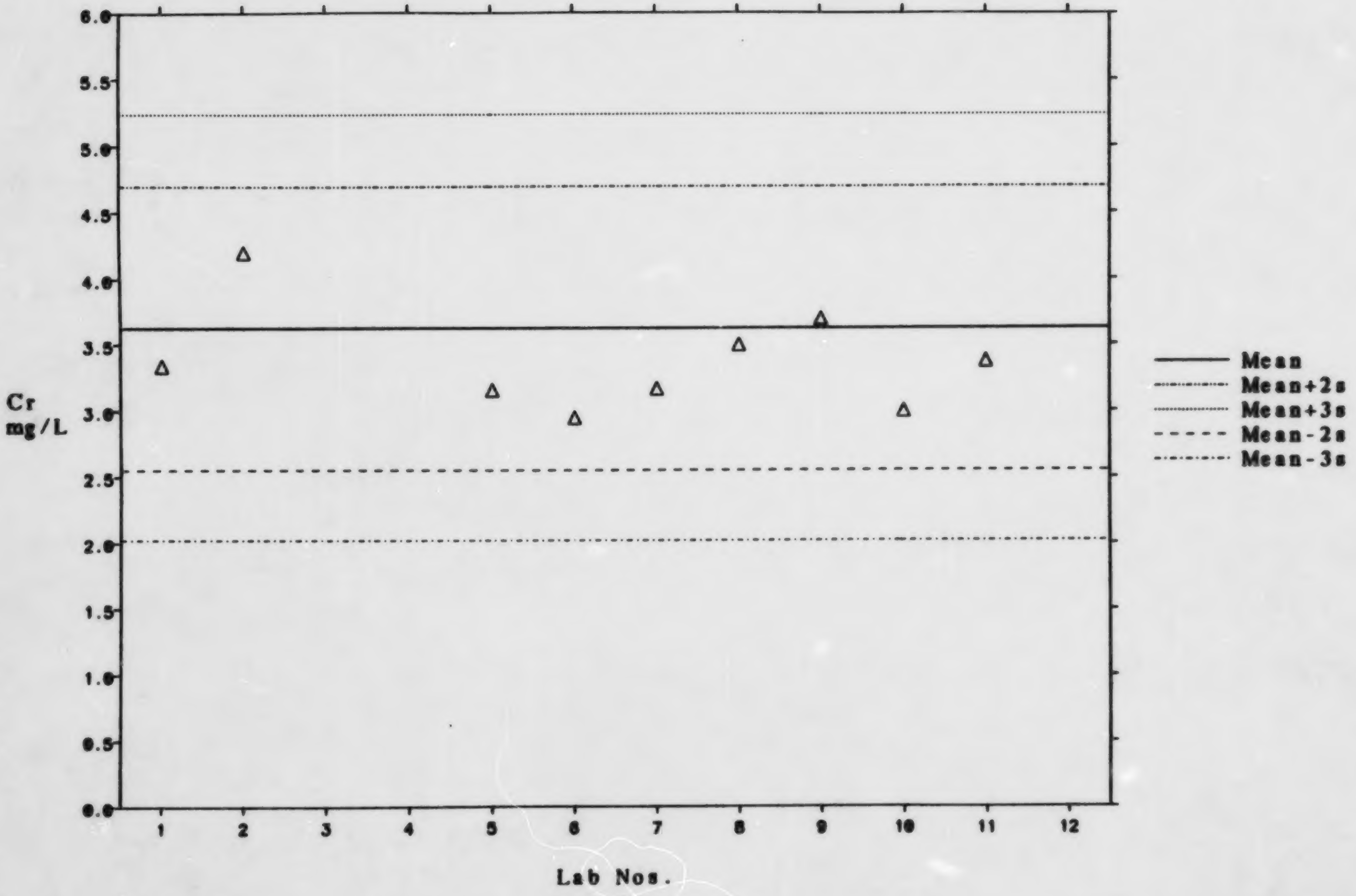


Figure 75. Mean and Confidence Intervals for Cr in Standard A

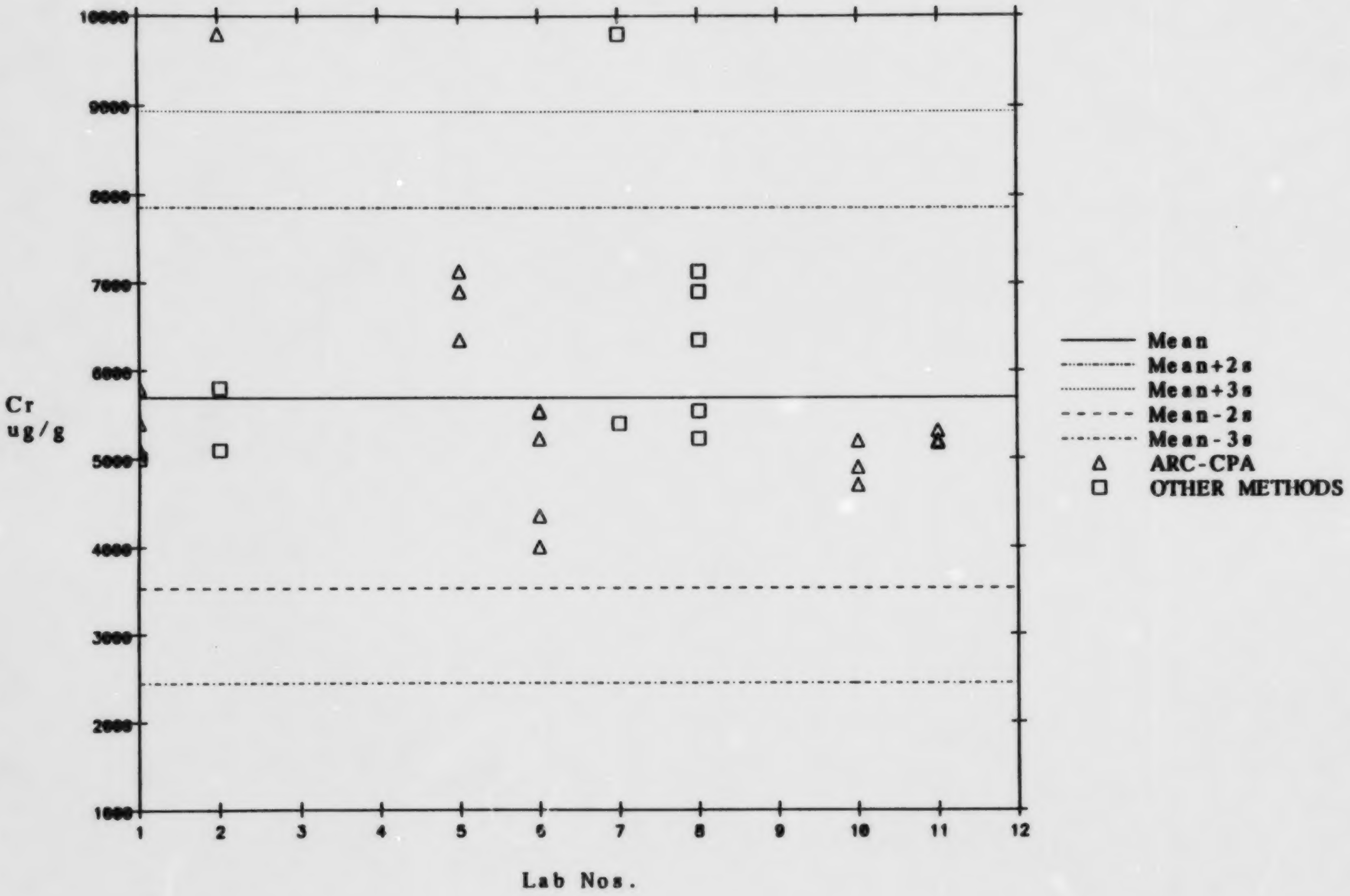


Figure 76. Mean and Confidence Intervals for Cr in Standard B

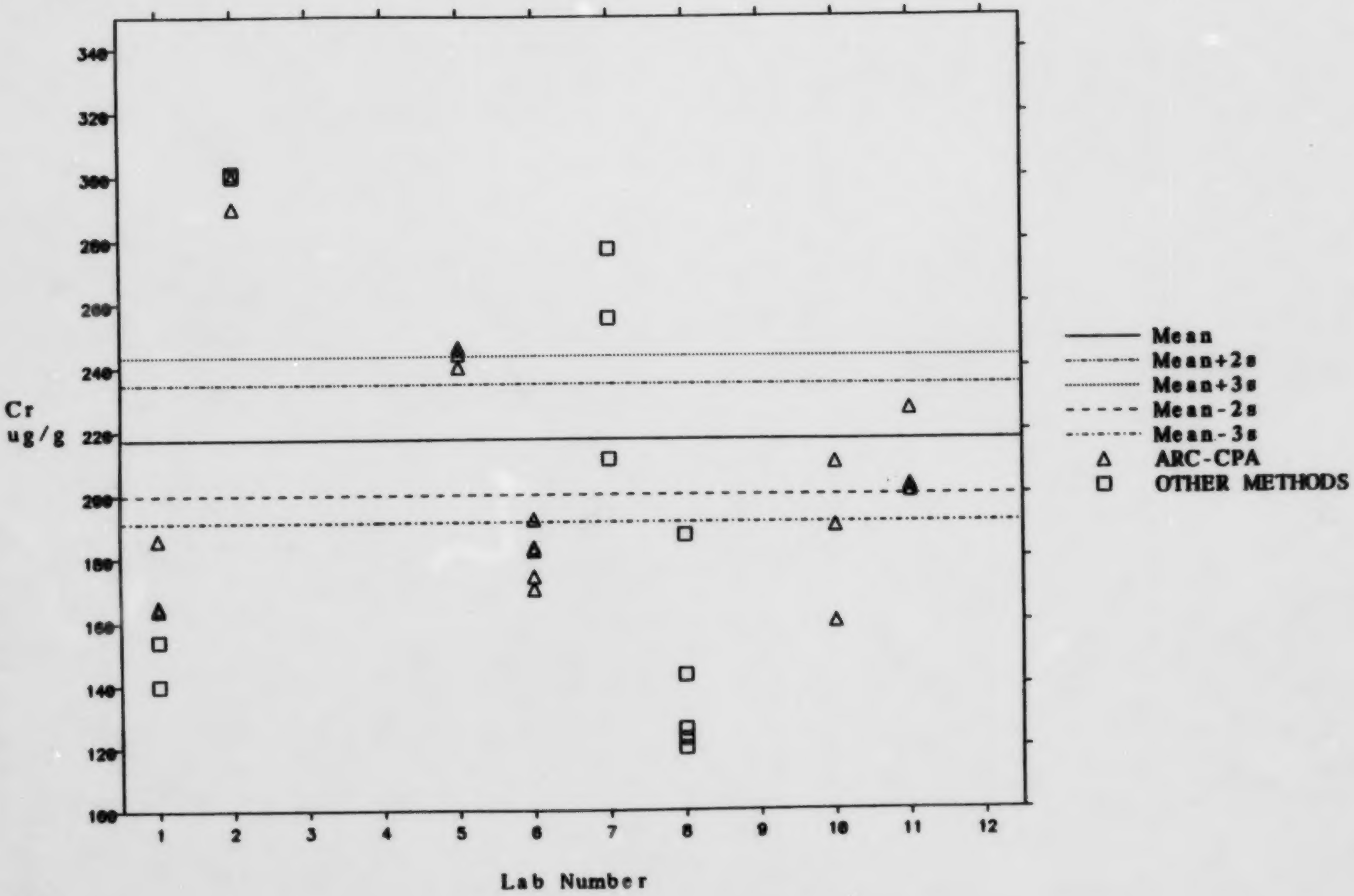


Table 9. Statistical Parameters for Cr (mg/Kg) in Sludges A-F

Statistic	Sludge					
	A	B	C	D	E	F
Count	36.00	41	35.0	42.0	35.0	34.0
Sum	968.00	11300	1430.0	4120.0	4670.0	1330.0
Mean	26.90	277	40.8	98.0	133.0	39.1
SEM	2.00	7	1.7	6.6	3.4	3.3
Median	26.00	267	40.0	85.8	129.0	30.7
Variance	143.0	1980	106.0	1840.0	393.0	373.0
StDev	12.00	45	10.3	42.9	19.8	19.3
Maximum	62.00	402	58.0	214.0	197.0	82.0
Minimum	0.64	210	10.0	42.0	111.0	17.0
Range	61.40	192	48.0	172.0	86.2	65.0
Skewness	1.10	7e-01	-0.6	1.0	1.6	0.8
Kurtosis	2.49	2e-01	1.1	0.3	2.5	-0.4
CV	44.50	16	25.2	43.7	14.9	49.5

SEM=Standard error of mean; StDev= Standard deviation;

C.V=Coefficient of variability;

Figure 77. Analysis of Cr in Sludge A

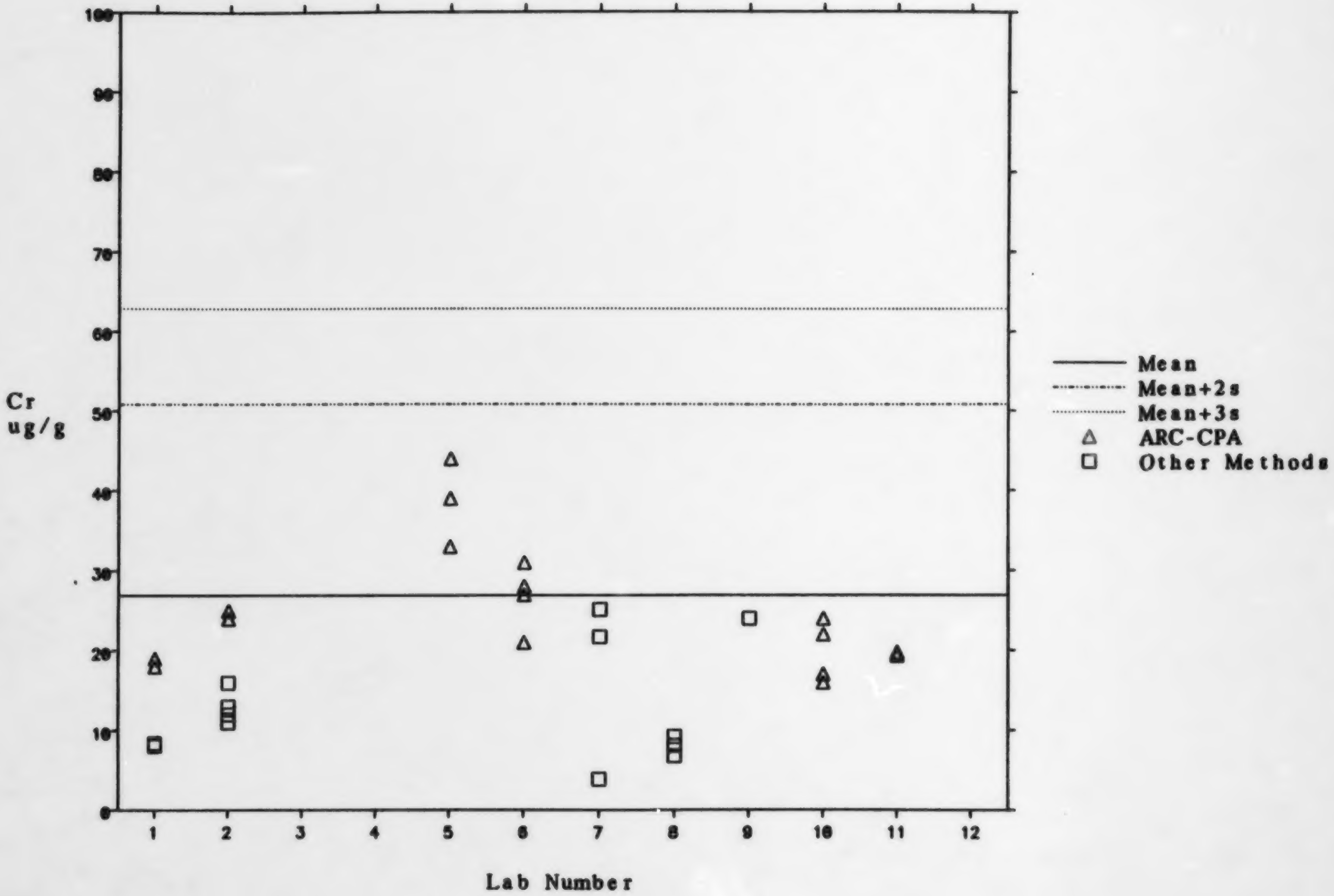


Figure 78. Analysis of Cr in Sludge B

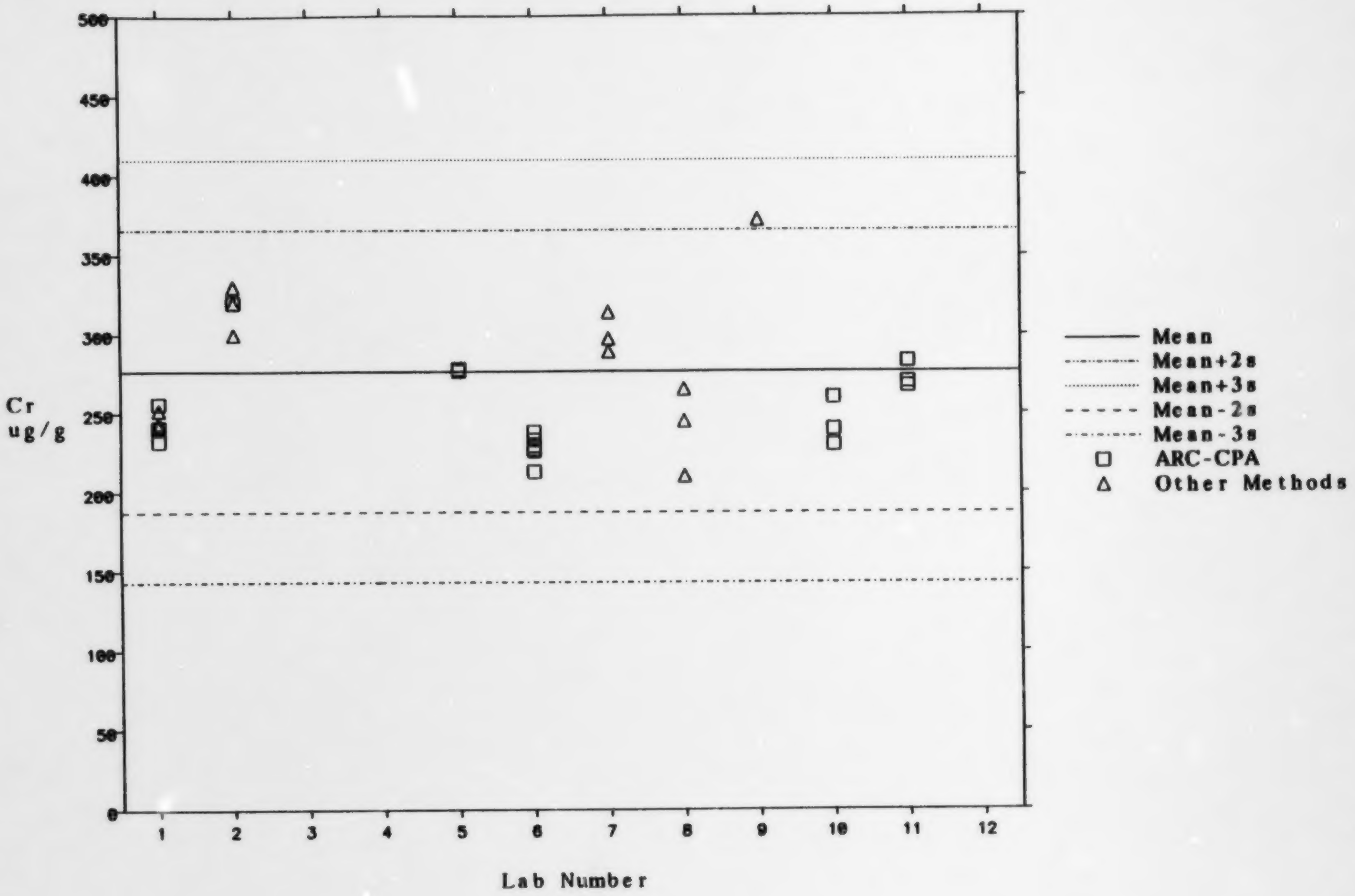


Figure 79. Analysis of Cr in Sludge C

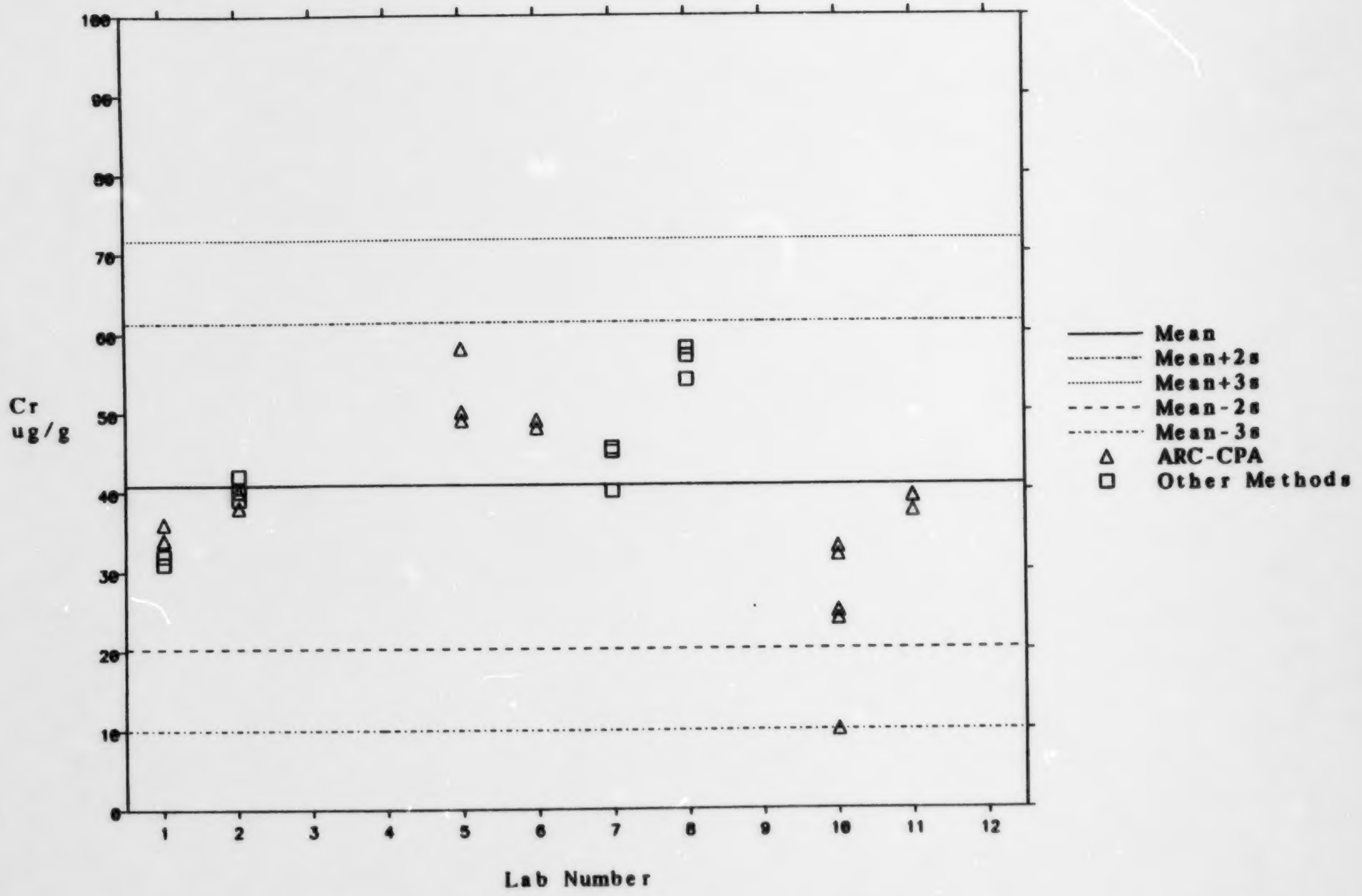


Figure 80. Analysis of Cr in Sludge D

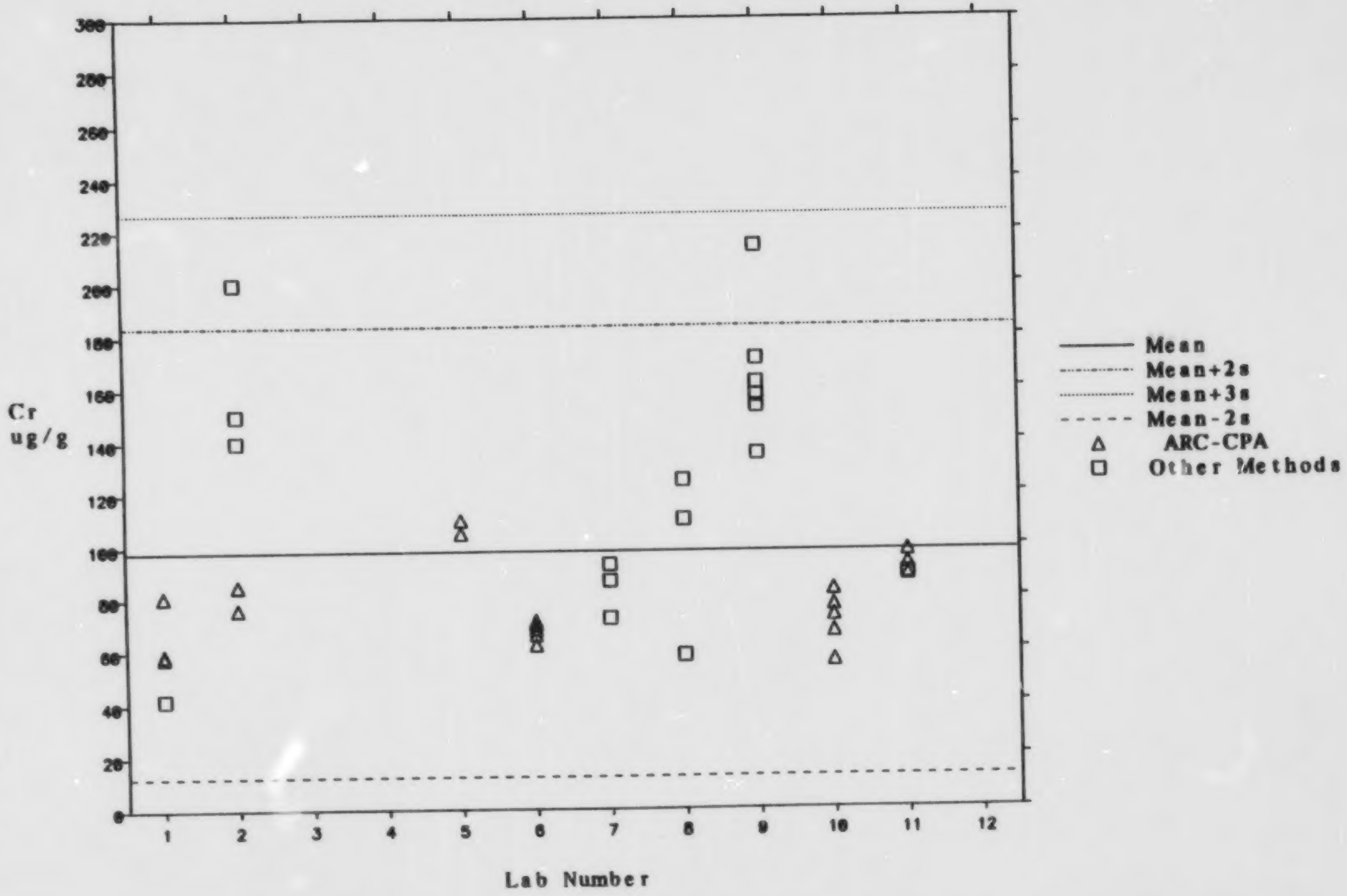


Figure 81. Analysis of Cr in Sludge E

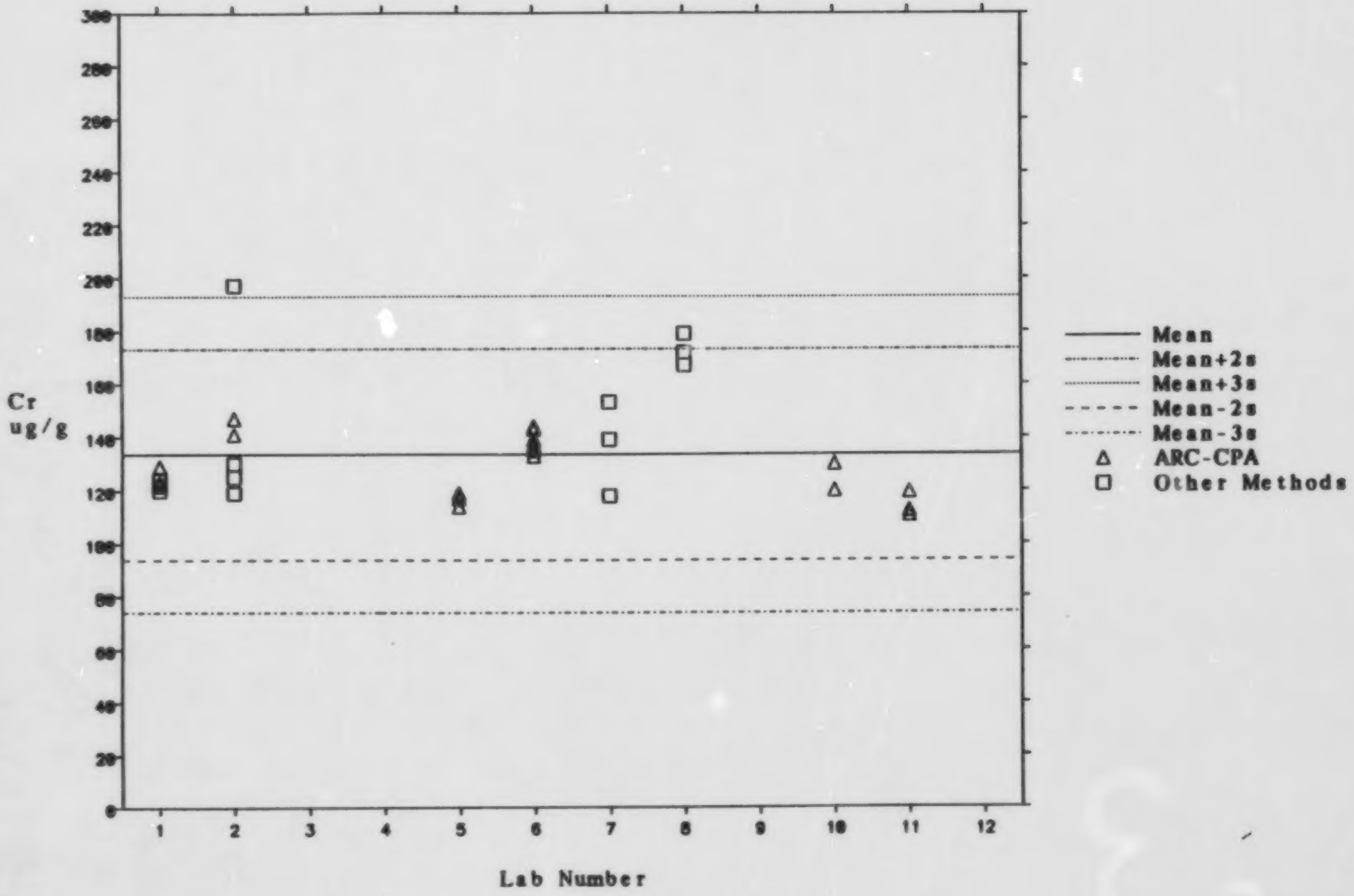
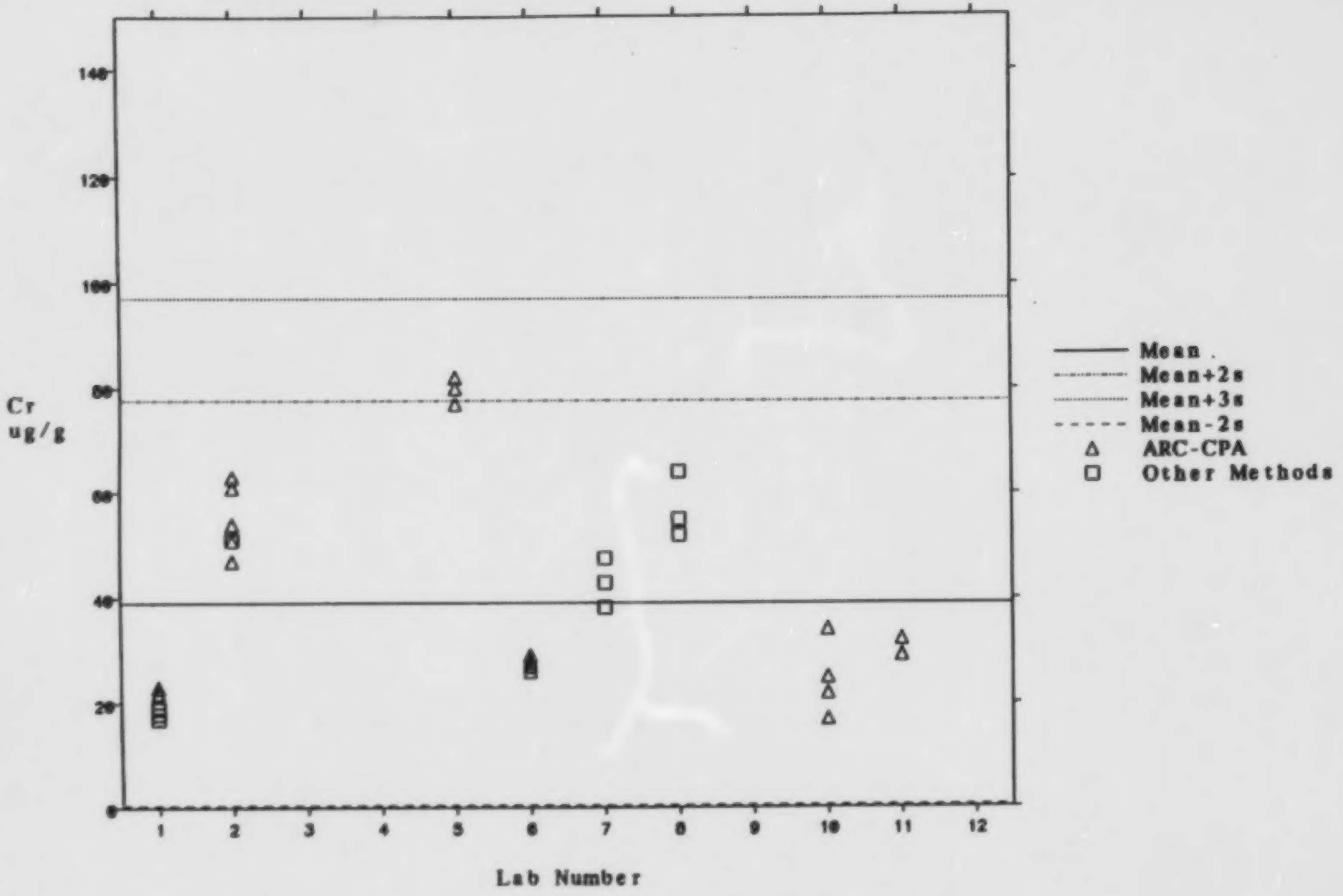


Figure 82. Analysis of Cr in Sludge F



dependent on the number of analyses performed and the Cr concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.1.9 Cu

Eight labs reported Cu in the ICP standard (Figure 83), all but one lab were within the control limits. Nine labs reported Cu for the digestate samples (Figures 84 and 85). Digestate 3 and 4 data (Figures 84 and 85) were all within the control limits, indicating close agreement between the labs and the actual digestate values. Eight labs reported Cu for the standard A and B samples (Figures 86 and 87). Figure 86 shows that all the reported data are within Standards A's warning limits. Standard B values (Figure 87) show that all but 3 labs had data within the warning limits, with 4 labs reporting some data both inside and outside the control limits. Figure 87 also shows that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Cu in sludges A to F are shown in Table 10, and their control charts in Figures 88 to 93. Table 10 shows that results (above detection) were reported for all sludges, with number of analyses varying from 4 to 42 samples. The mean Cu values ranged from 7.5 mg/kg for sludge E to 258 for sludge B. The coefficient of variation of the sludges varied from 11 to 48% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 88 to 93 indicate that all the reported Cu data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Cu data shows good recovery of the ICP, digestate and standard samples. The data also shows some agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed and the Cu concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

Figure 83. Mean and Confidence Intervals for Cu in an ICP Standard

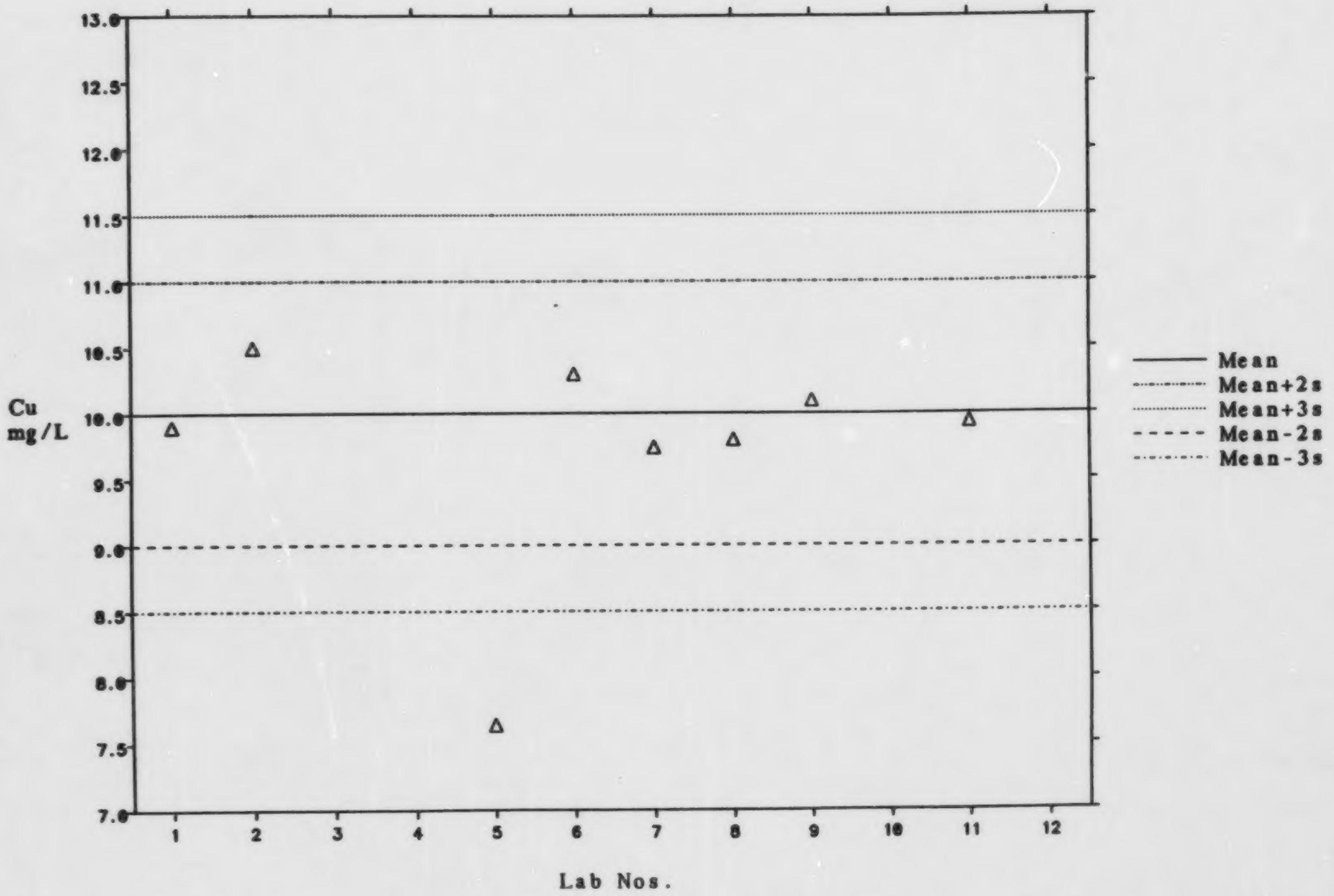


Figure 84. Mean and Confidence Intervals for Cu in Digestate 3

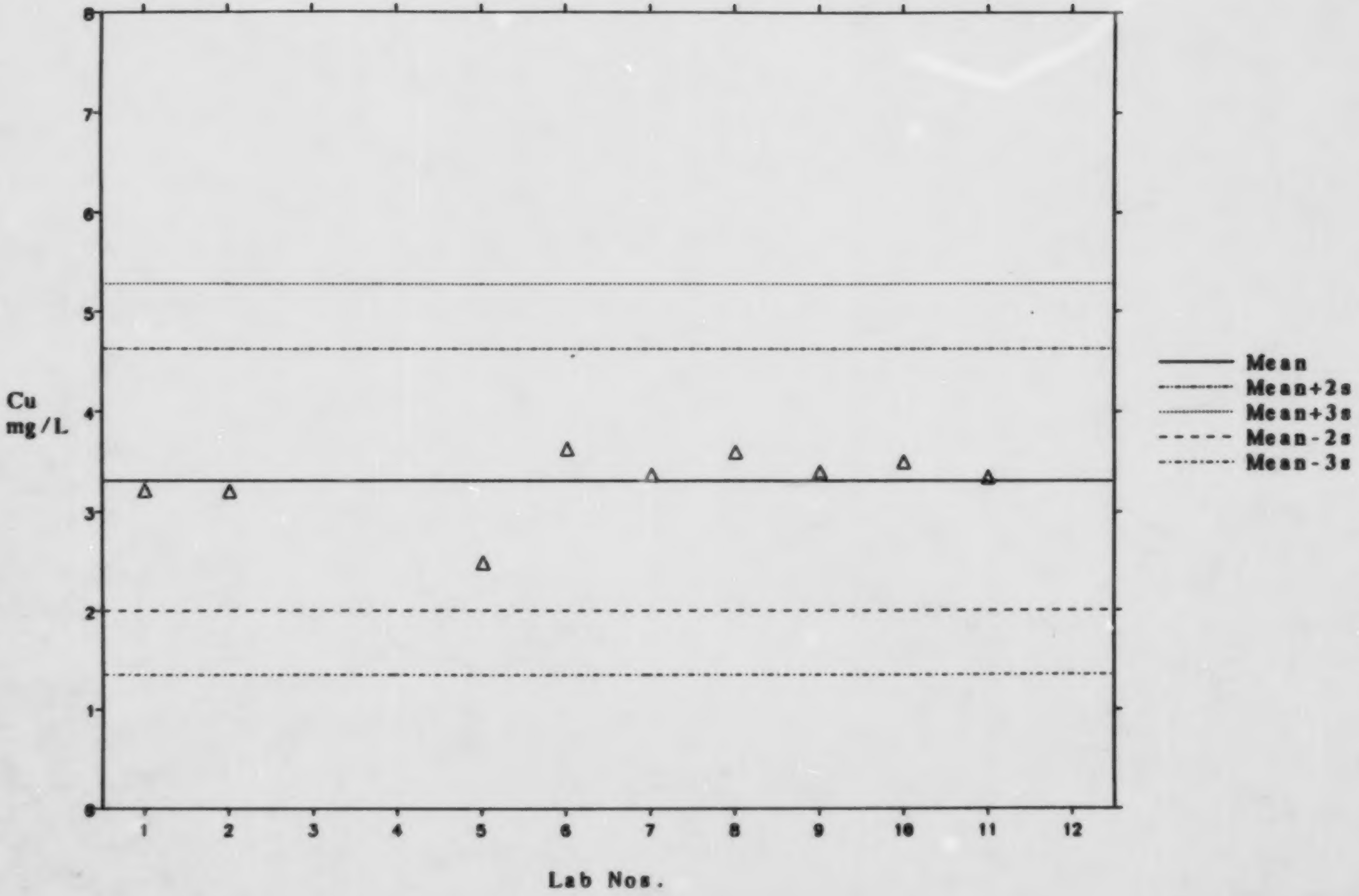


Figure 85. Mean and Confidence Intervals for Cu in Digestate 4

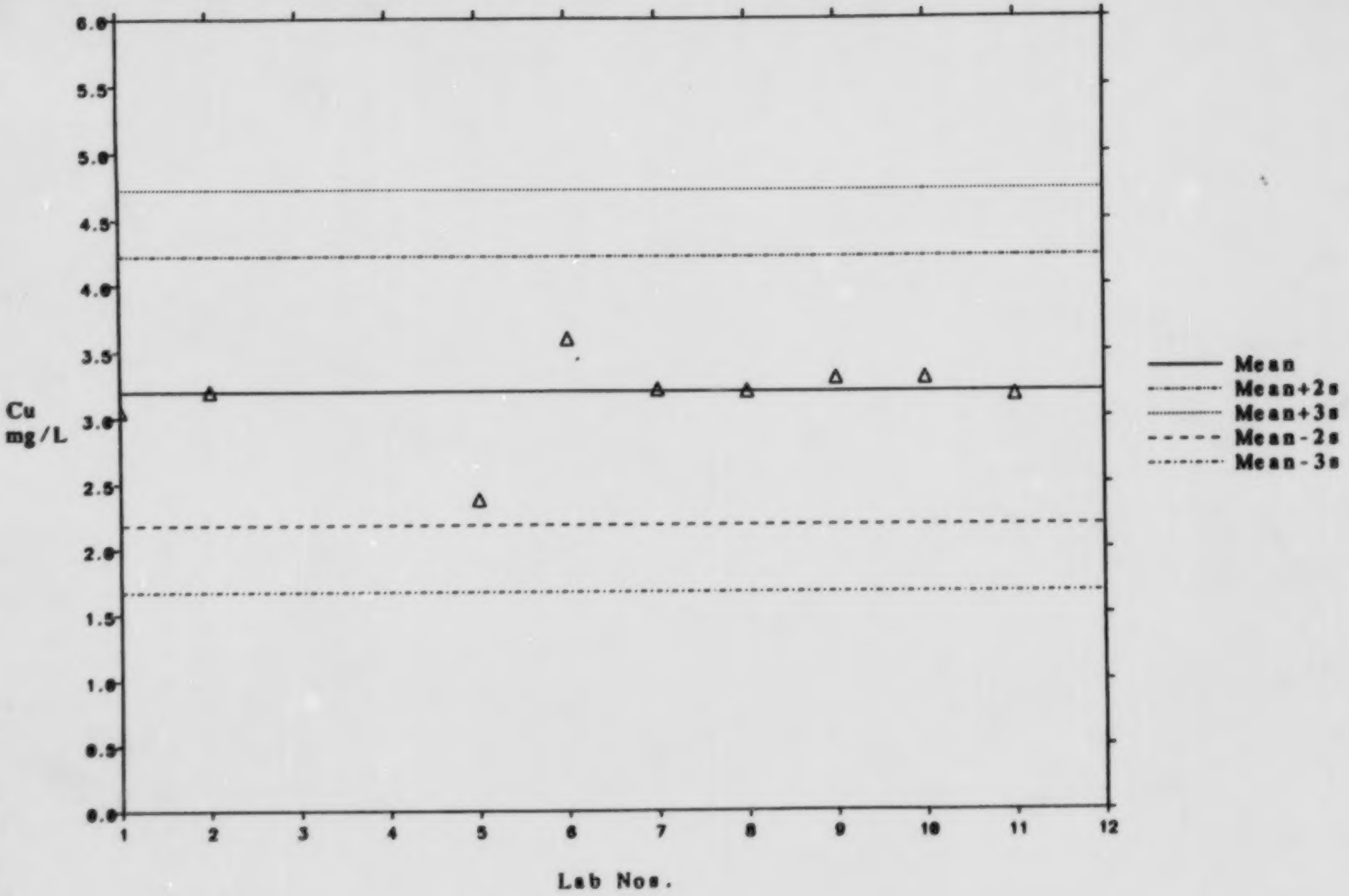


Figure 86. Mean and Confidence Intervals for Cu in Standard A

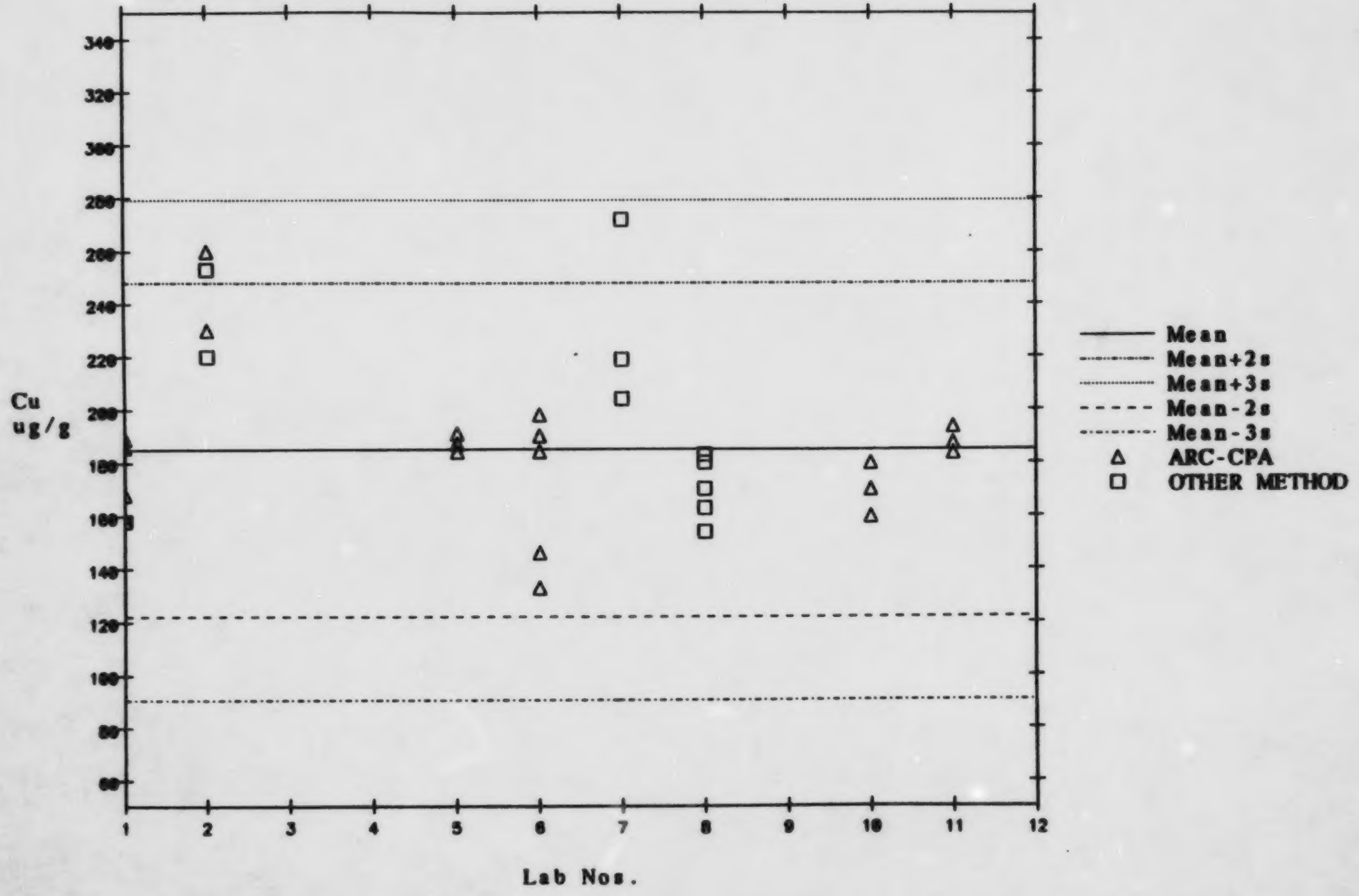


Figure 87. Mean and Confidence Intervals for Cu in Standard B

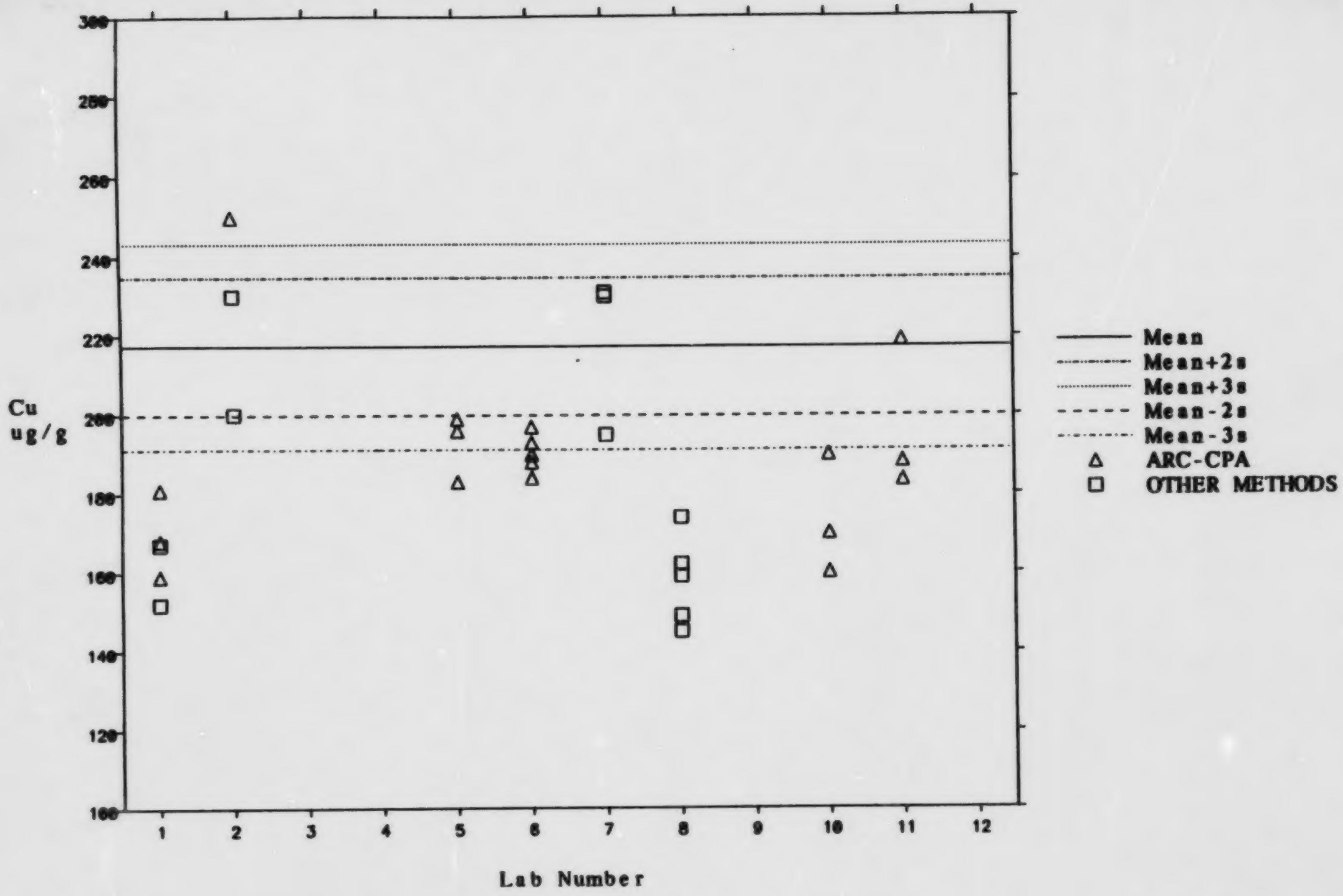


Table 10. Statistical Parameters for Cu (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	36.00	41	35.0	42.0	4.00	34.0
Sum	416.00	10600	2010.0	1770.0	30.00	1490.0
Mean	11.50	258	57.5	42.2	7.50	43.9
SEM	0.92	4	1.5	2.2	1.76	2.3
Median	11.00	258	57.0	40.5	7.50	39.0
Variance	30.70	787	79.4	202.0	12.30	182.0
StDev	5.54	28	8.9	14.2	3.51	13.5
Maximum	25.10	330	72.0	87.0	11.00	92.0
Minimum	2.00	205	39.0	23.2	4.00	29.5
Range	23.10	125	33.0	63.8	7.00	62.5
Skewness	0.78	3e-02	-0.3	1.1	0.00	1.7
Kurtosis	0.61	7e-02	-0.3	1.4	-5.21	3.5
CV(%)	48.00	11	15.5	33.7	46.80	30.7

SEM=Standard error of mean; StDev= Standard deviation;

C.V=Coefficient of variability;

Figure 88. Analysis of Cu in Sludge A

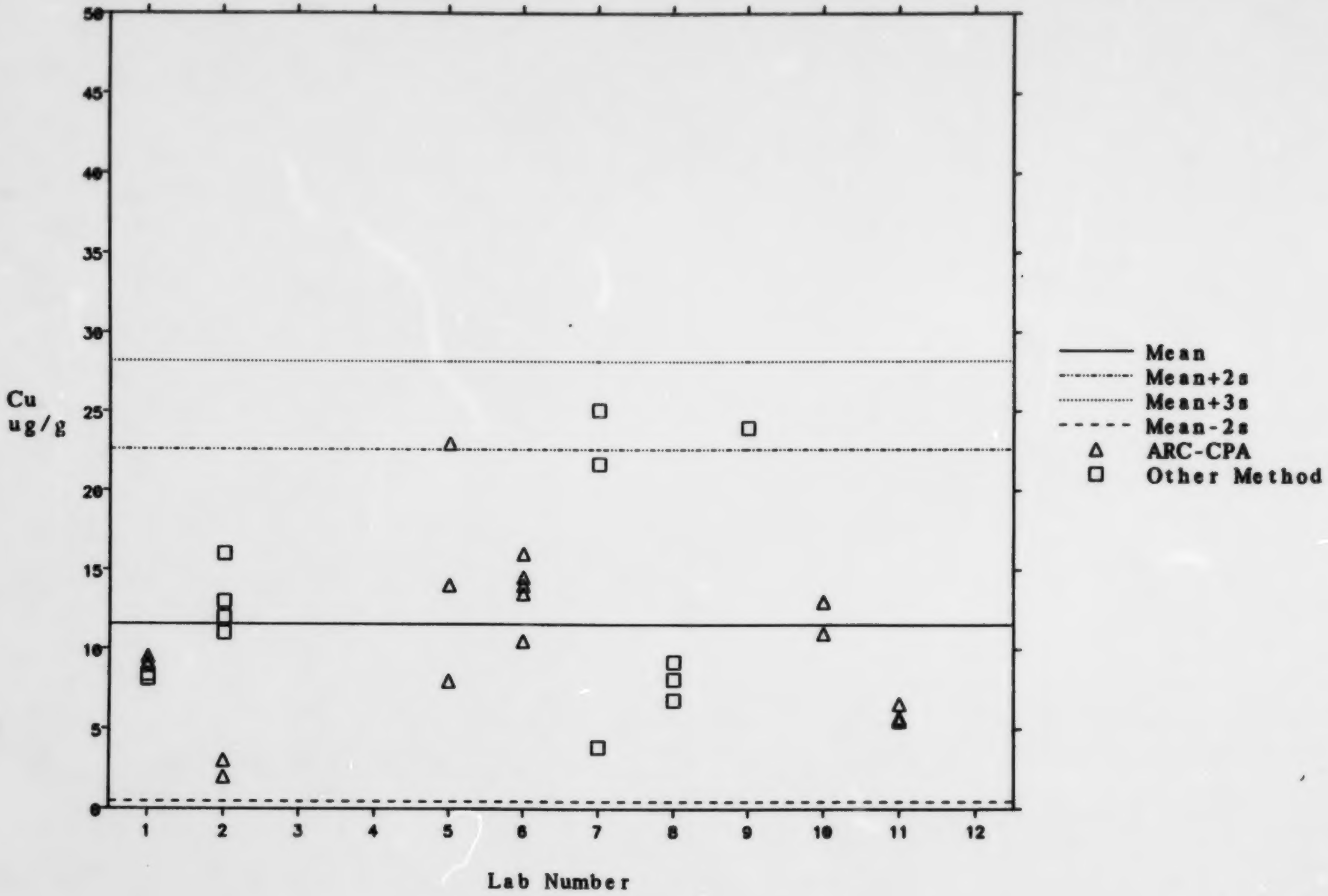


Figure 89. Analysis of Cu in Sludge B

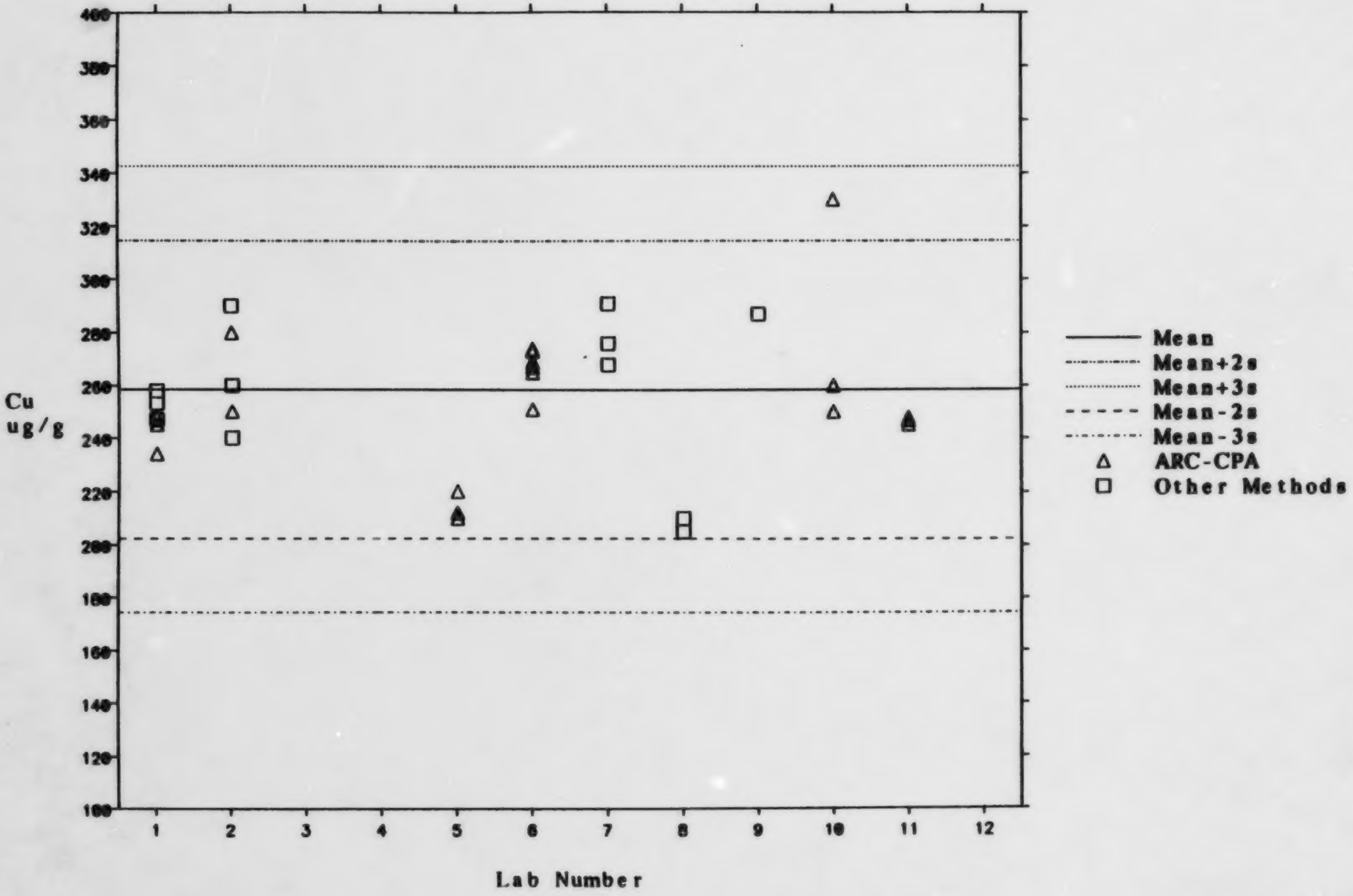


Figure 90. Analysis of Cu in Sludge C

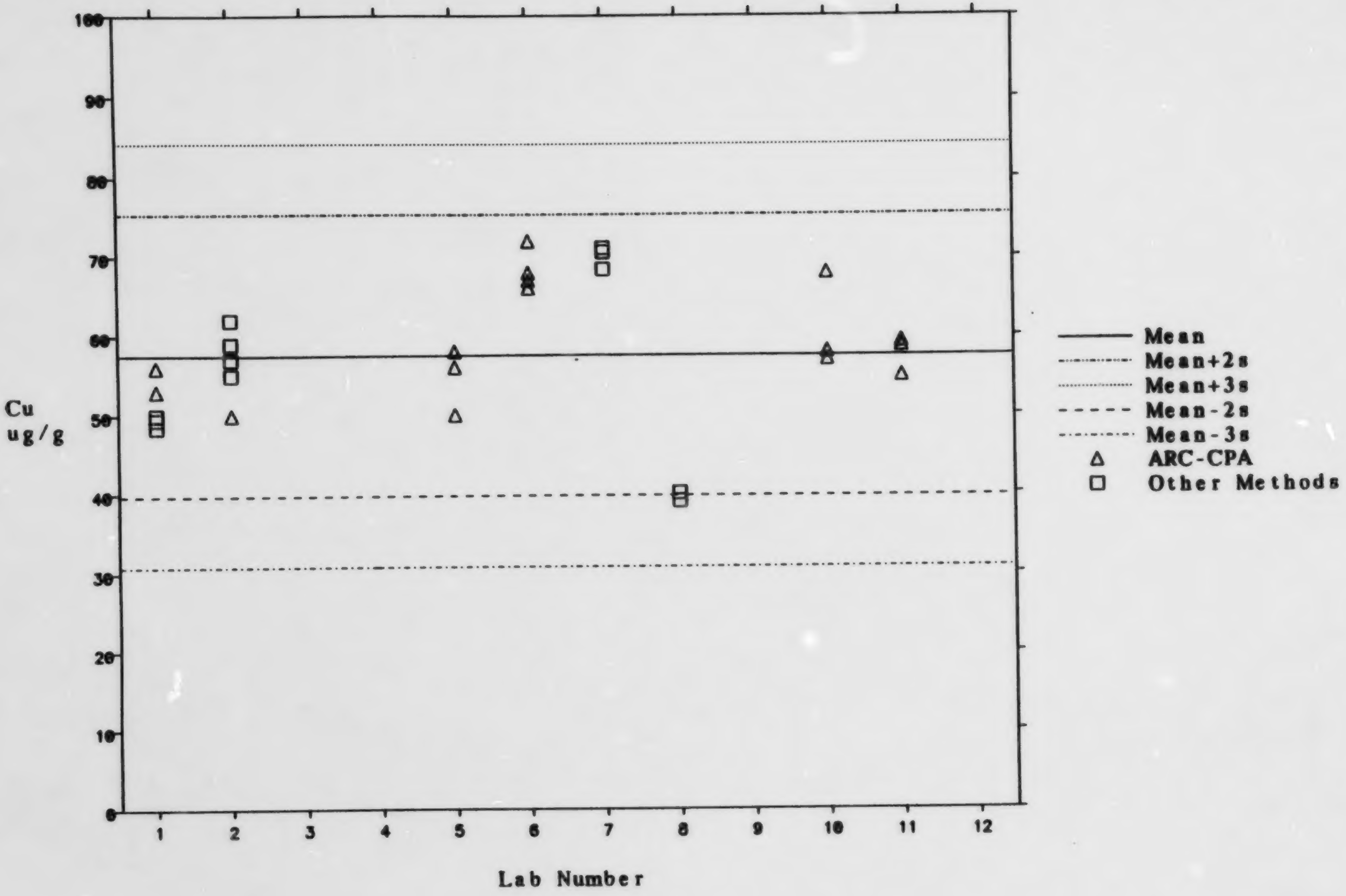


Figure 91. Analysis of Cu in Sludge D

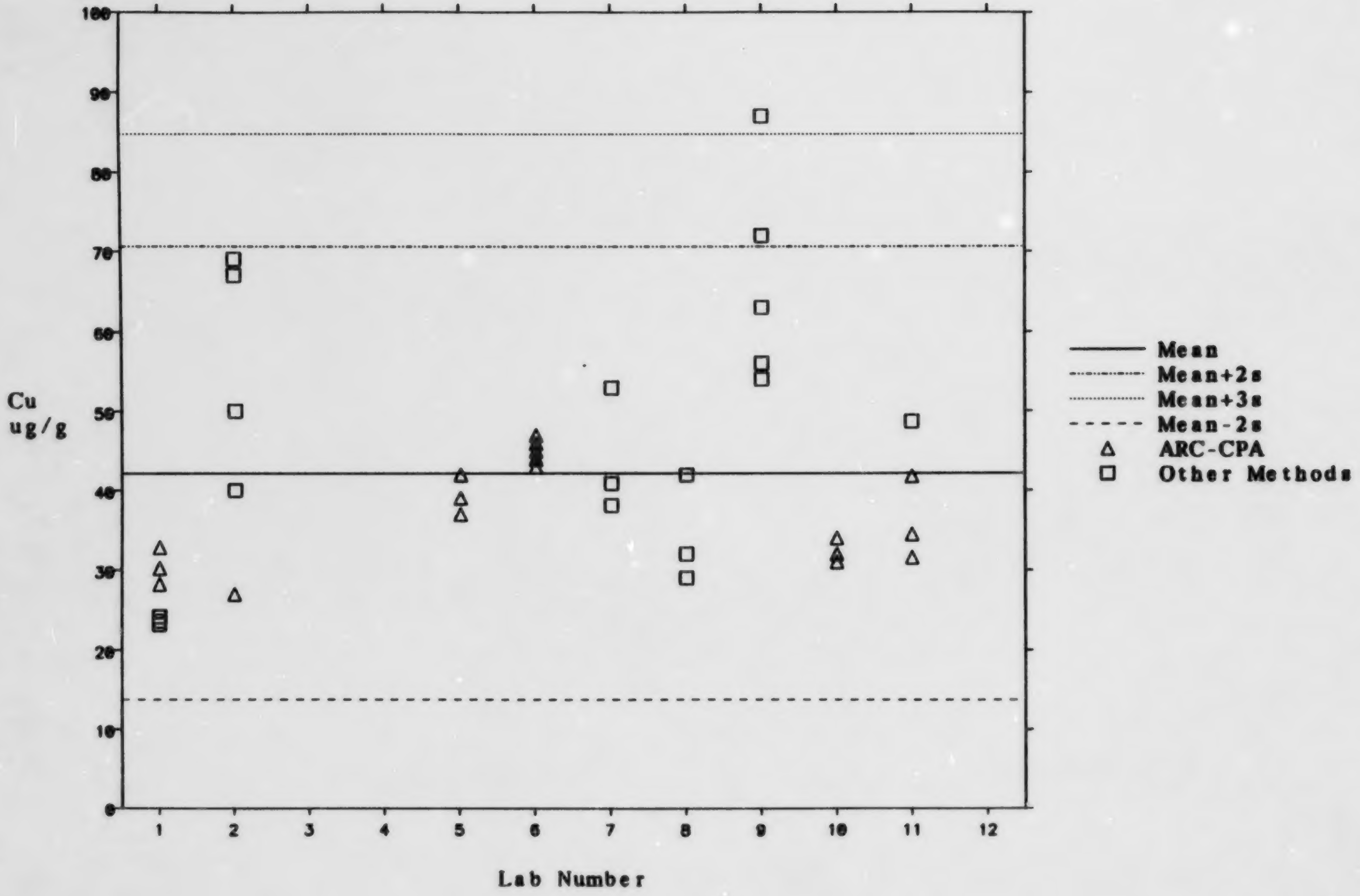


Figure 92. Analysis of Cu in Sludge E

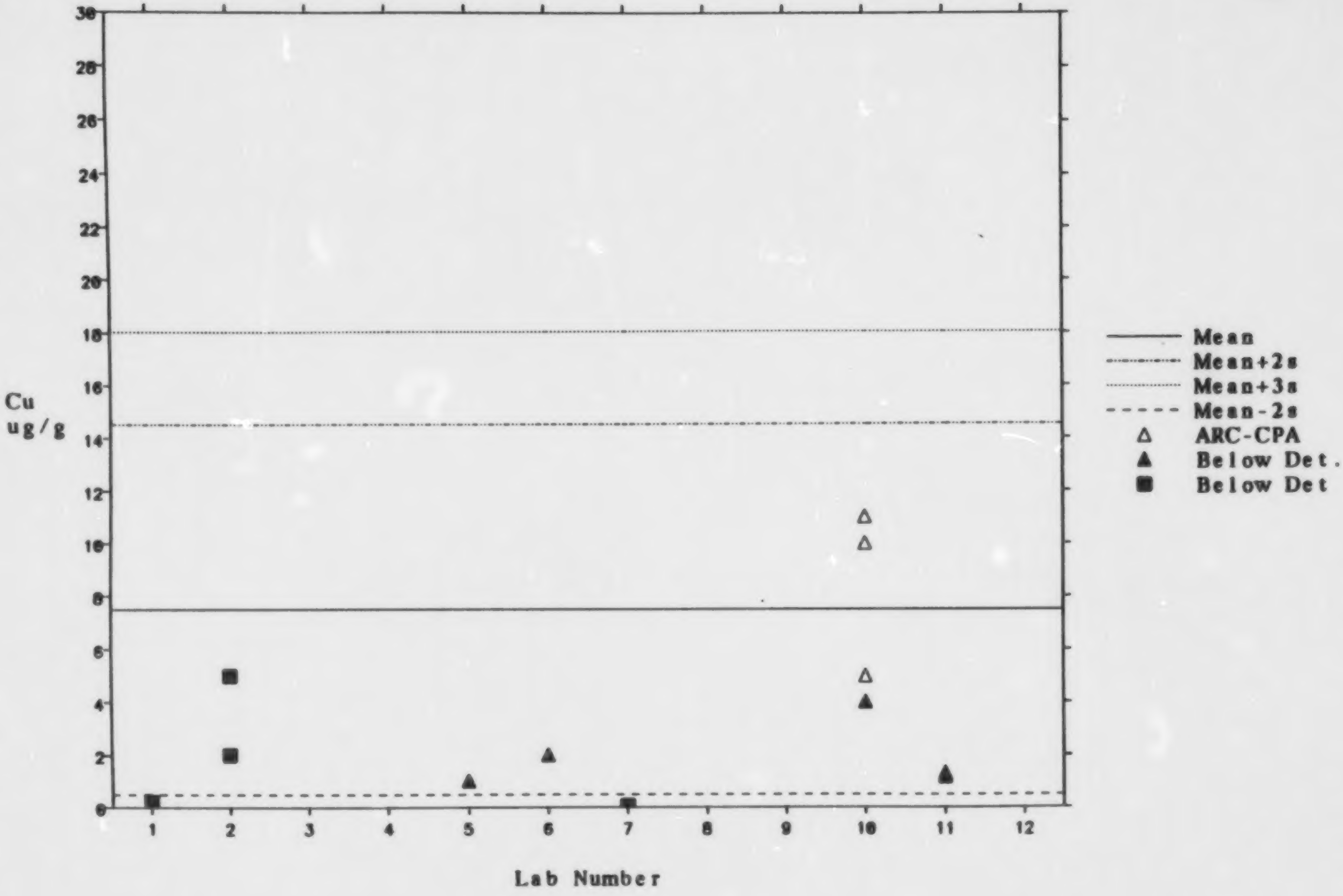
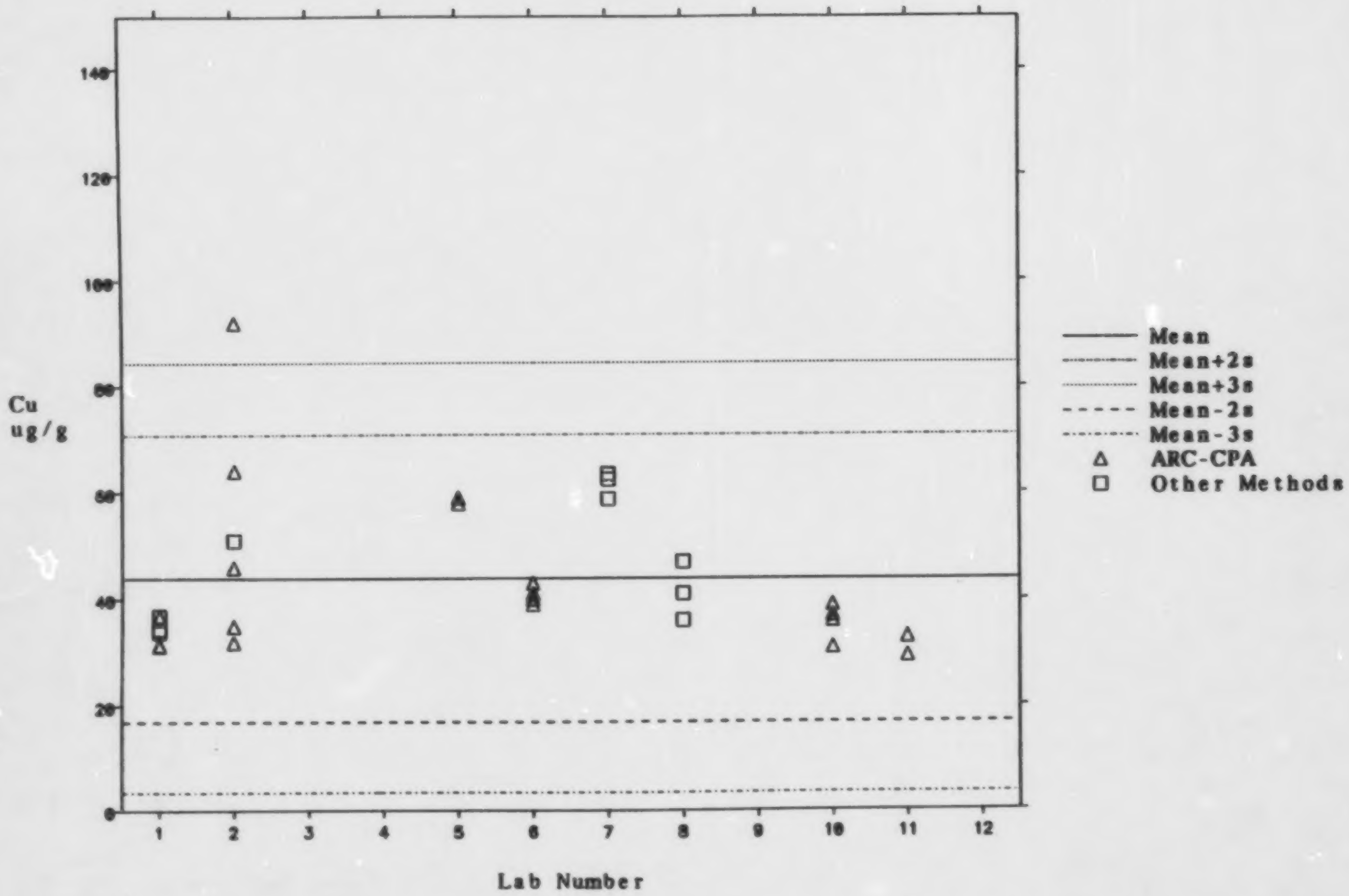


Figure 93. Analysis of Cu in Sludge F



4.1.1.10 Hg

Three labs reported Hg in the ICP standard (Figure 94), all were outside the control limits. Two labs reported Hg for the digestate samples (Figures 95 and 96). Digestate 3 and 4 data (Figures 95 and 96) show that one lab was within and one outside the control limits. Three labs reported Hg for the standard A and B samples (Figures 97 and 98). Figure 97 shows that the 3 labs reported data within Standards A's control limits. Standard B values (Figure 98) show that all labs are outside the warning limits. The differences between Standards A and B seem to be due to their different standard deviation, and thus, warning and control limits. Figures 97 and 98 also show that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Hg in sludges A to F are shown in Table 11, and their control charts in Figures 99 to 104. Table 11 shows that results (above detection) were reported for all sludges, with number of analyses varying from 7 to 10 samples. The mean Hg values ranged from 0.2 mg/kg for sludge E to 68 for sludge B. The coefficient of variation of the sludges varied from 87 to 181% and it does not seem to be related to the number of reported results or the measured concentrations.

Figures 99 to 104 indicate that all the reported Hg data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Hg data is difficult to interpret due to the low number of analyzed samples and participating labs.

4.1.1.11 Mn

Six labs reported Mn in the ICP standard (Figure 105), all but two labs were within the control limits. The two outlier labs were both lower than the control warning limit. Seven labs reported Mn for the digestate samples (Figures 106 and 107). Digestate 3 and 4 data

Figure 94. Mean and Confidence Intervals for Hg in an ICP Standard

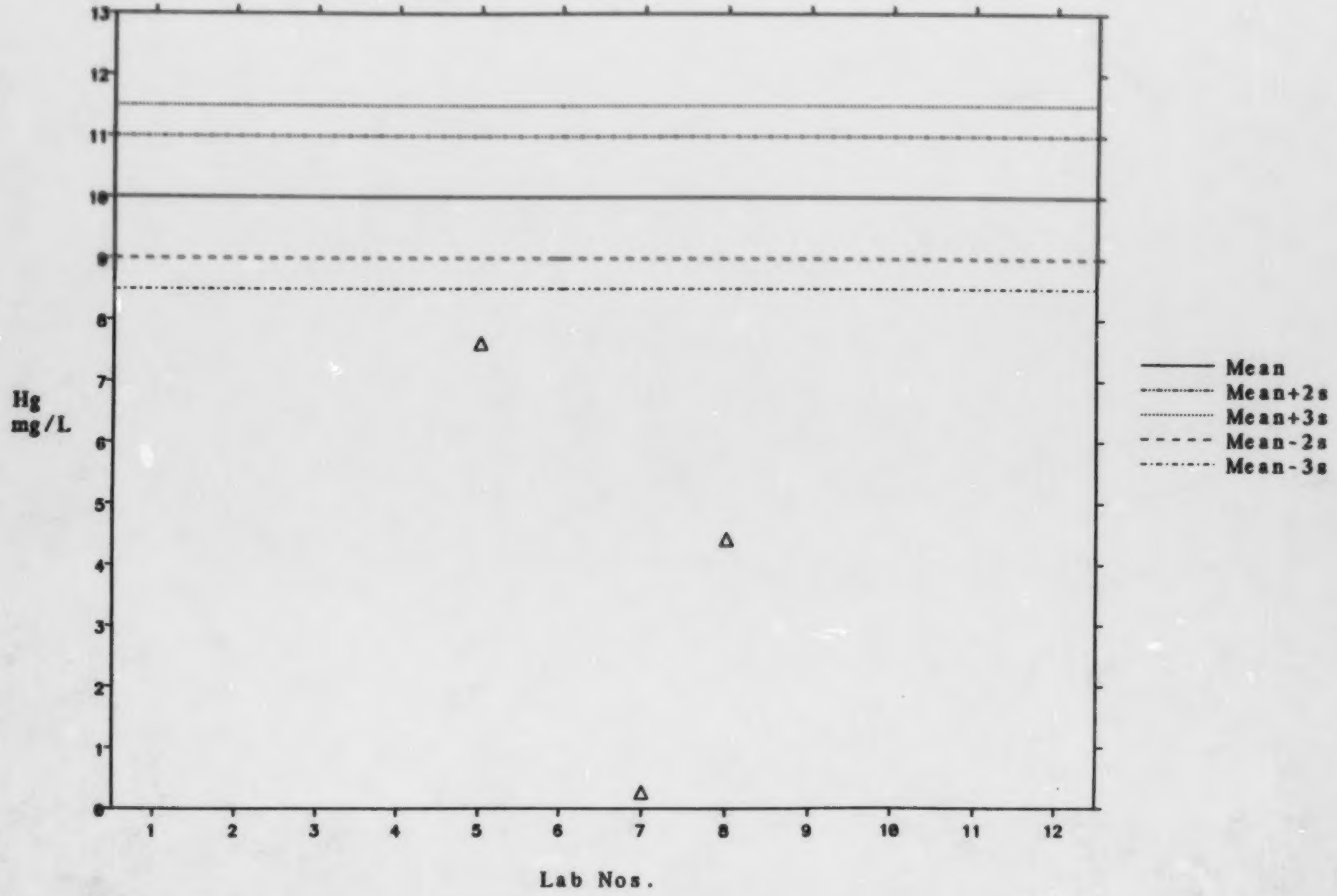


Figure 95. Mean and Confidence Intervals for Hg in Digestate 3

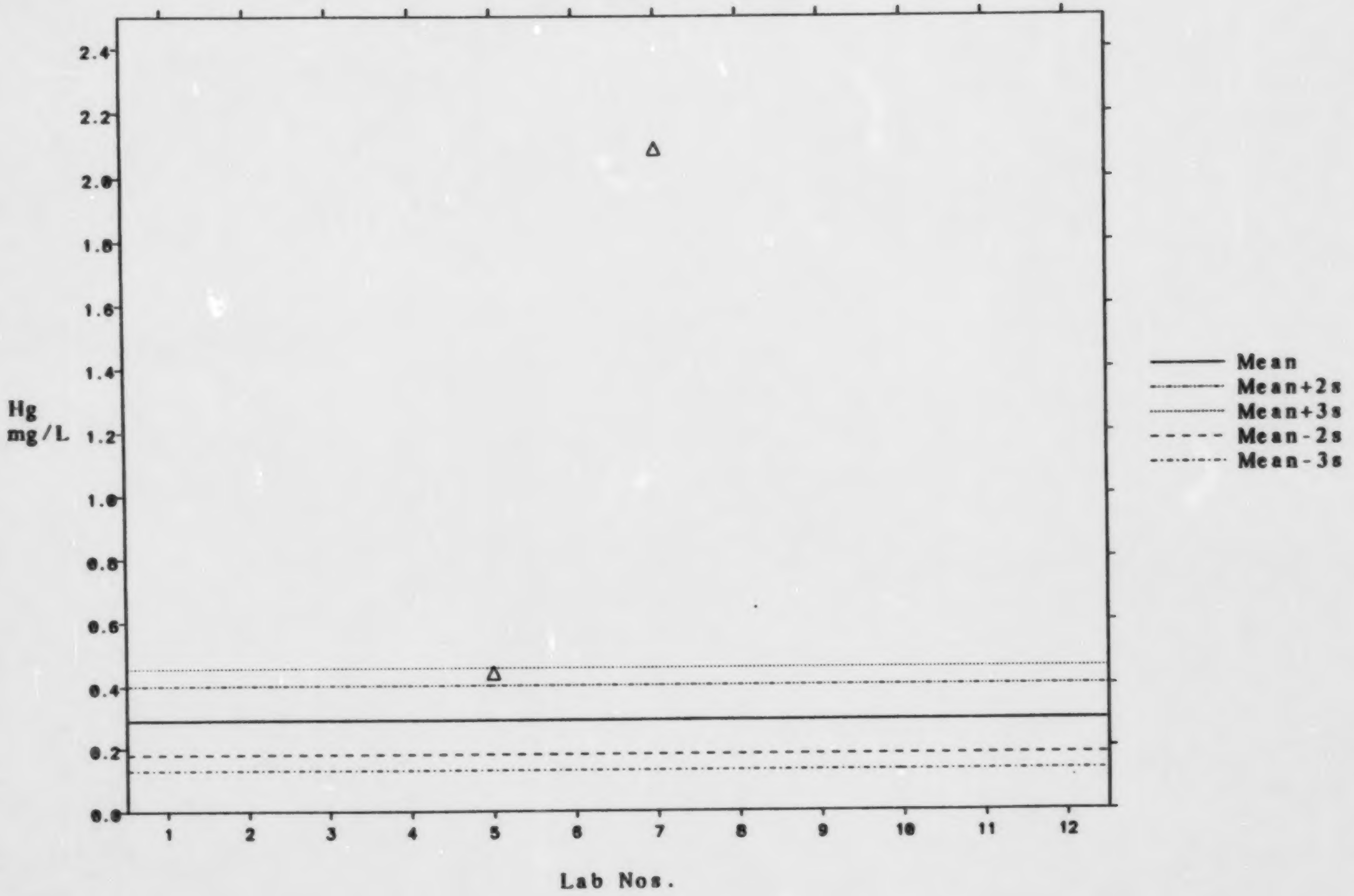


Figure 96. Mean and Confidence Intervals for Hg in Digestate 4

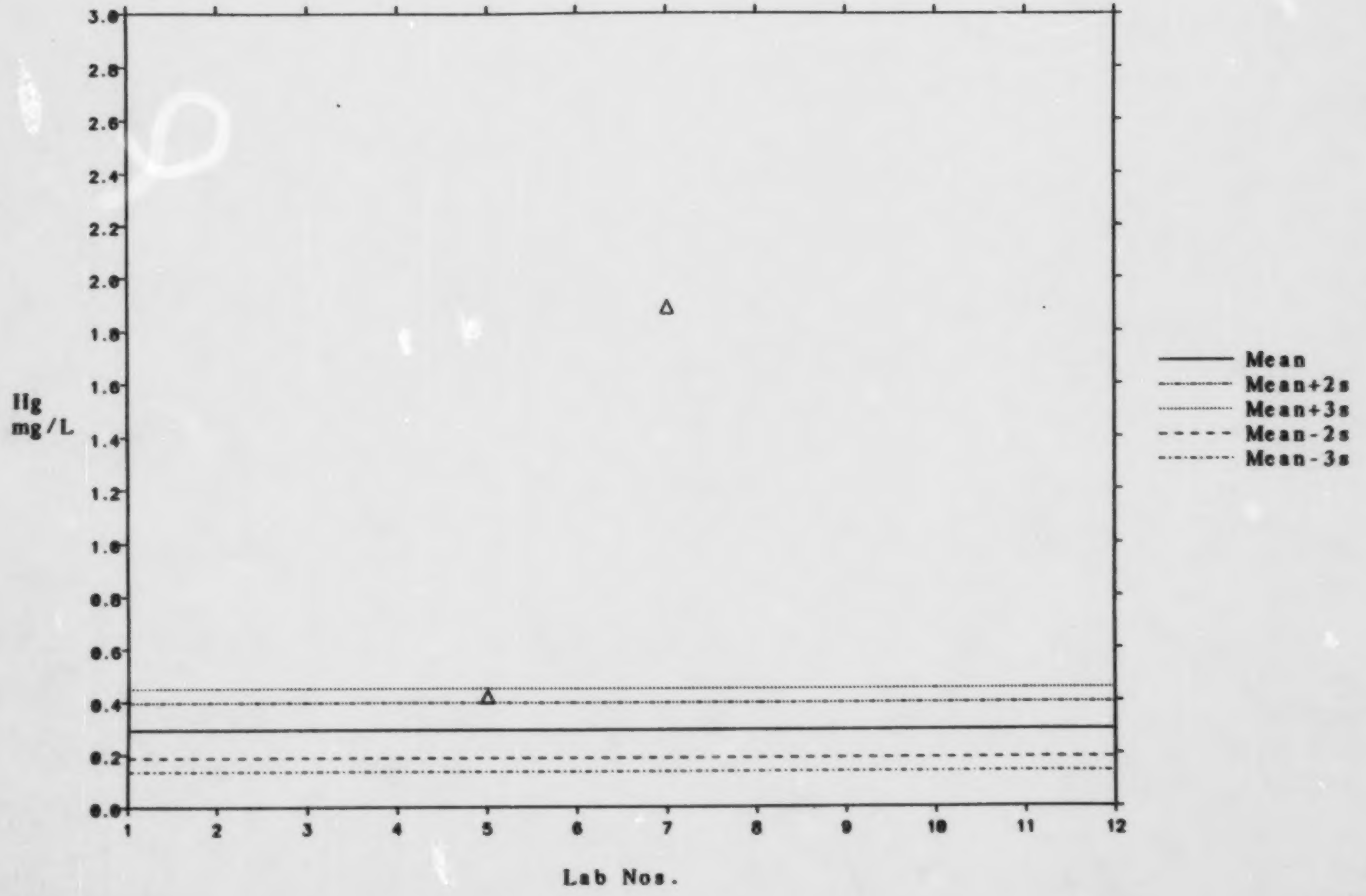


Figure 97. Mean and Confidence Intervals for Hg in Standard A

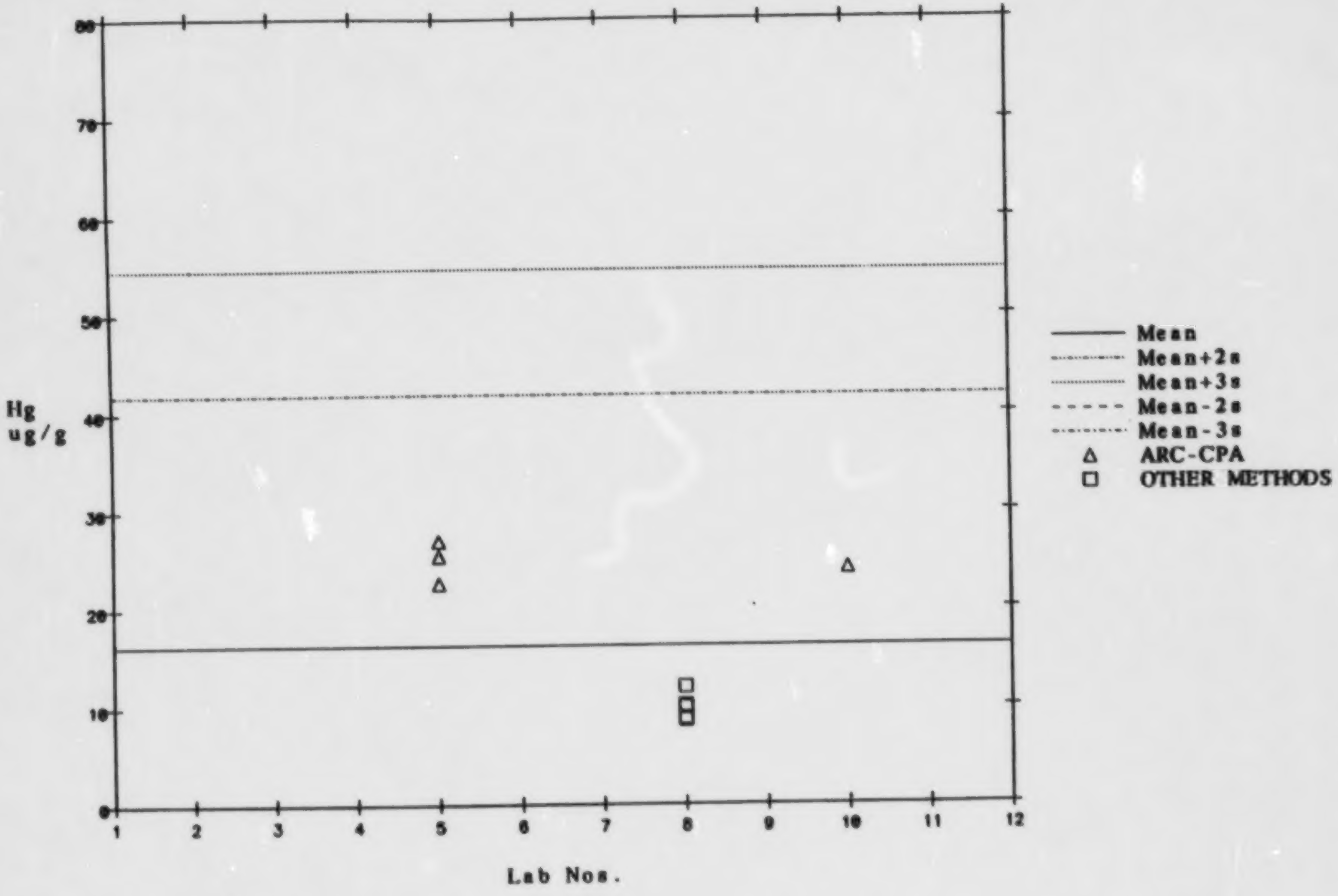


Figure 98. Mean and Confidence Intervals for Hg in Standard B

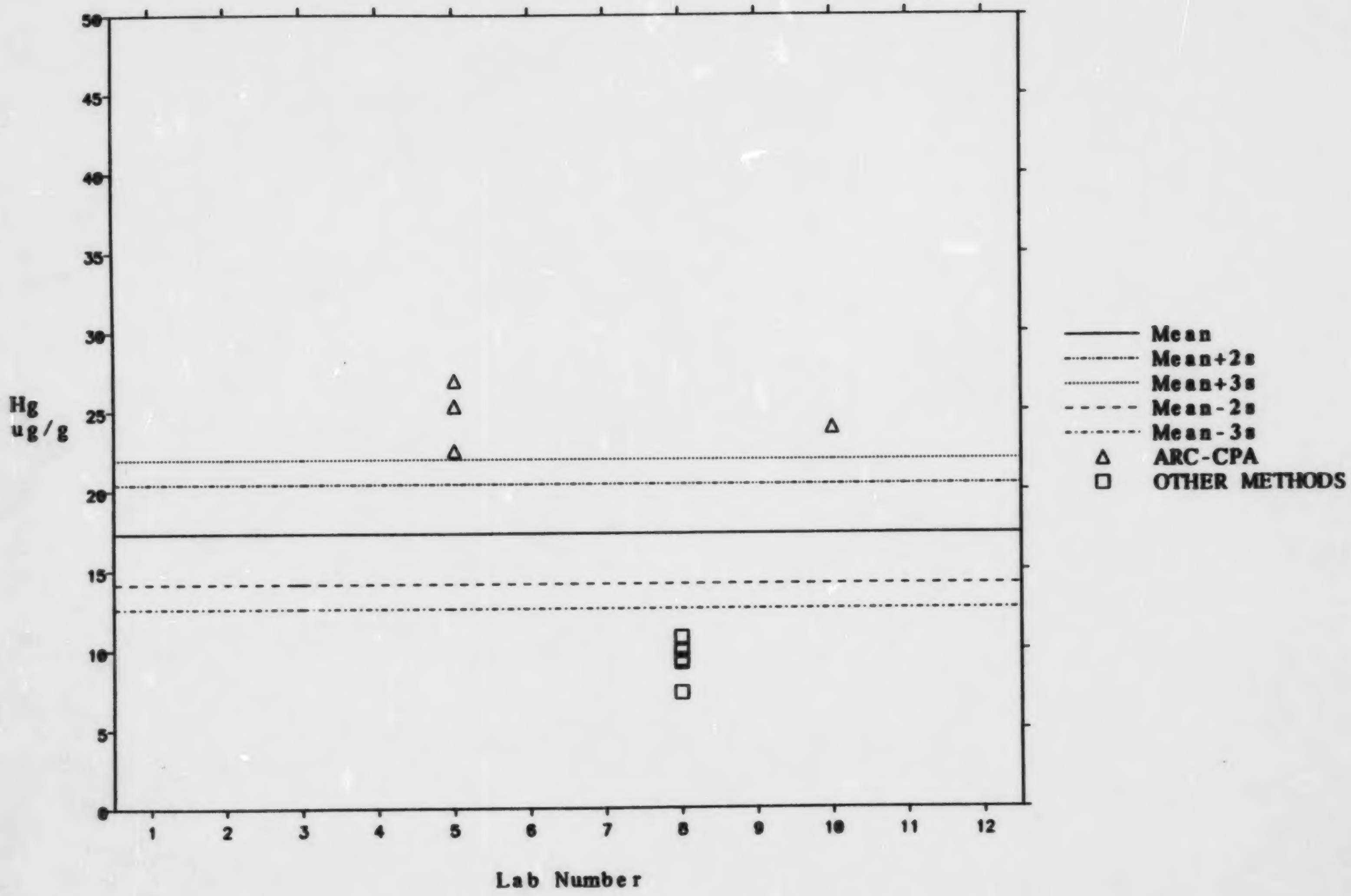


Table 11. Statistical Parameters for Hg (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	10.00	10.0	10.00	10.00	7.000	10.0
Sum	56.70	680.0	56.70	185.00	1.370	319.0
Mean	5.67	68.0	5.67	18.50	0.196	31.9
SEM	3.24	28.2	2.32	8.76	0.065	17.5
Median	1.27	24.6	2.21	2.87	0.250	2.8
Variance	105.0	7930.0	53.60	767.00	0.029	3060.0
StDev	10.20	89.0	7.32	27.70	0.171	55.3
Maximum	27.20	236.0	17.20	65.20	0.450	166.0
Minimum	0.12	0.4	0.23	0.22	0.020	8e-02
Range	27.10	236.0	17.00	65.00	0.430	166.0
Skewness	1.82	1.3	1.00	1.12	0.202	1.9
Kurtosis	1.71	-3e-02	-1.18	-0.80	-1.570	3.5
CV(%)	181.00	131.0	129.00	150.00	87.200	173.0

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 99. Analysis of Hg in Waste A

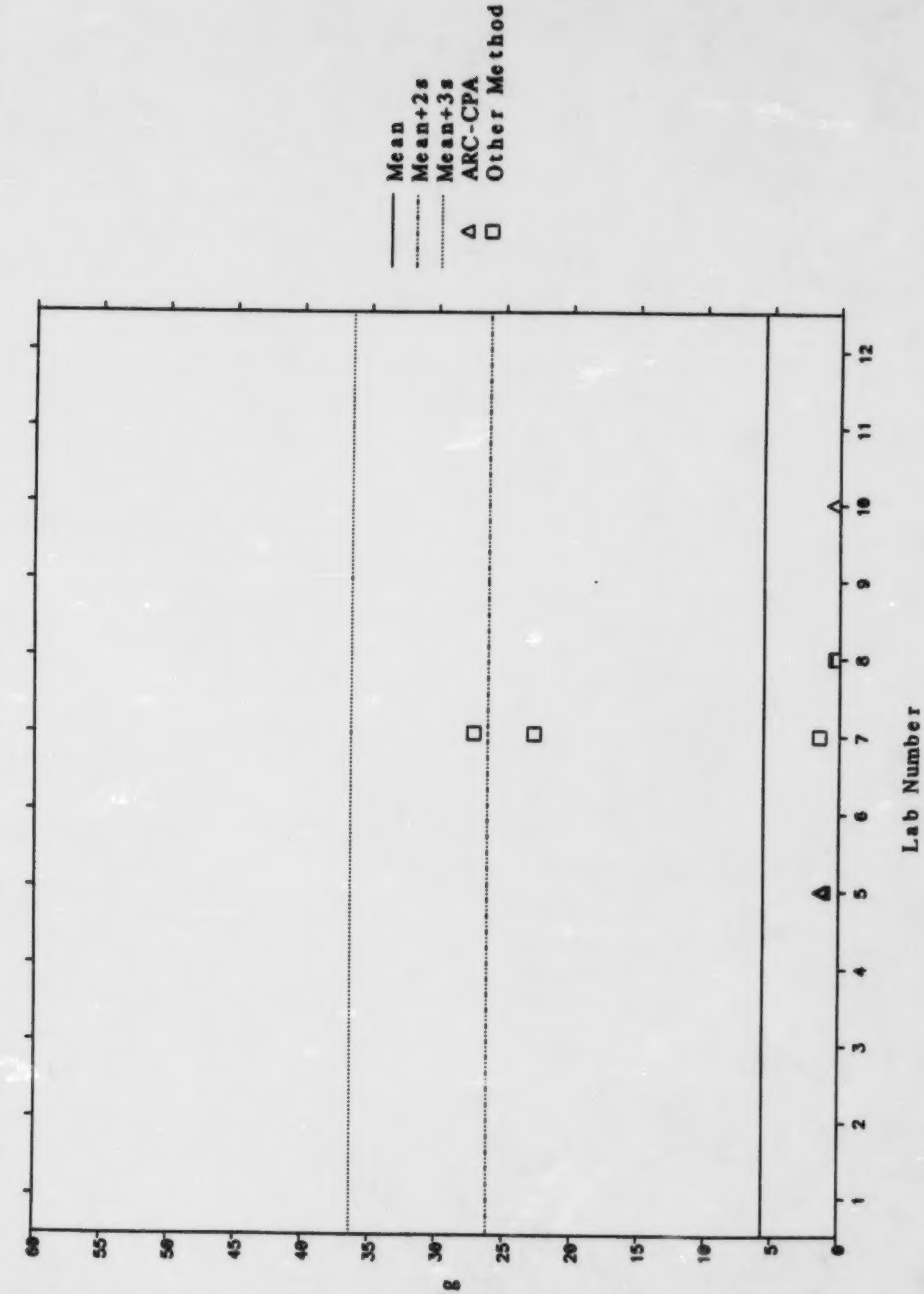


Figure 100. Analysis of Hg in Sludge B

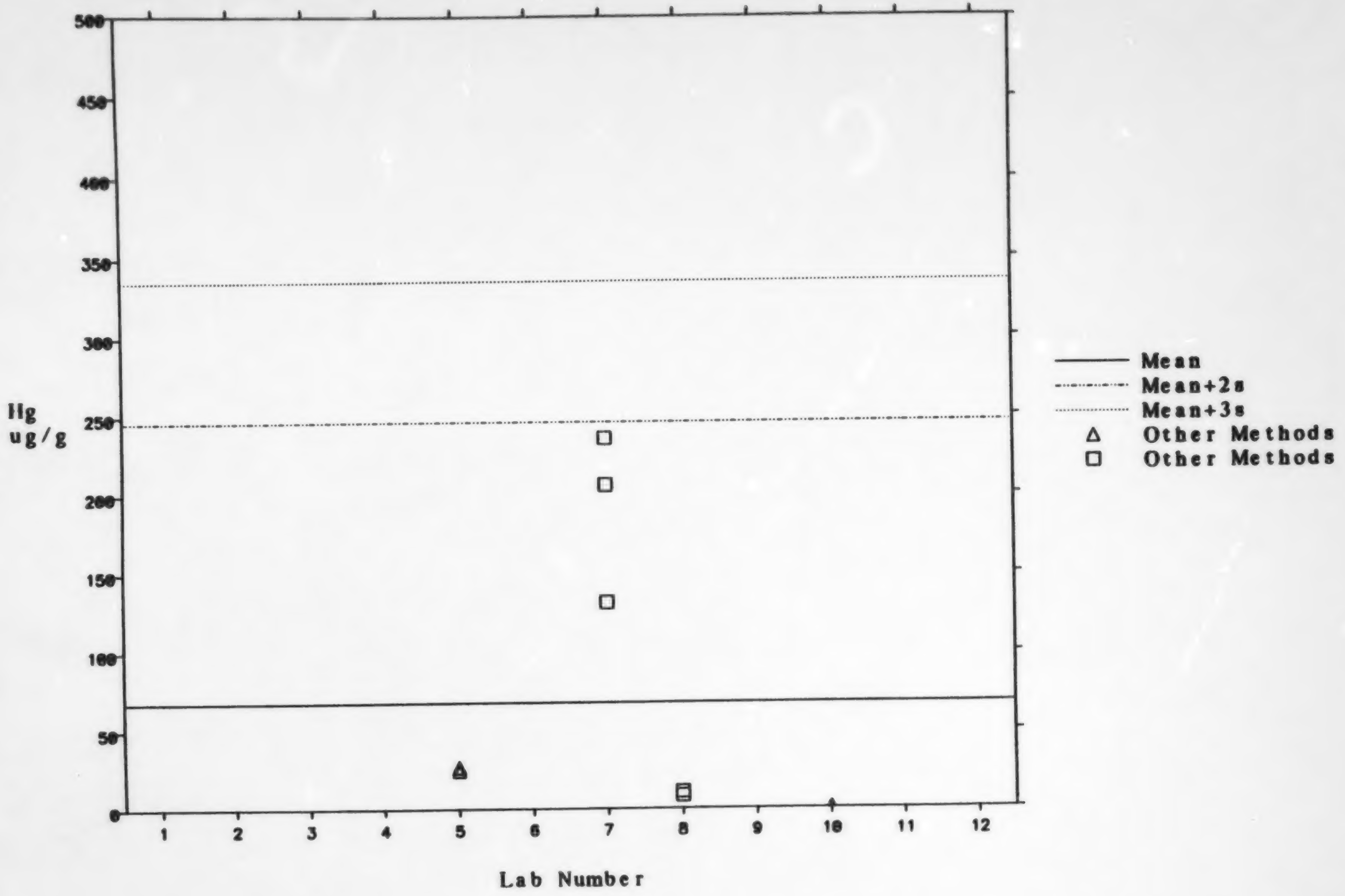


Figure 101. Analysis of Hg in Sludge C

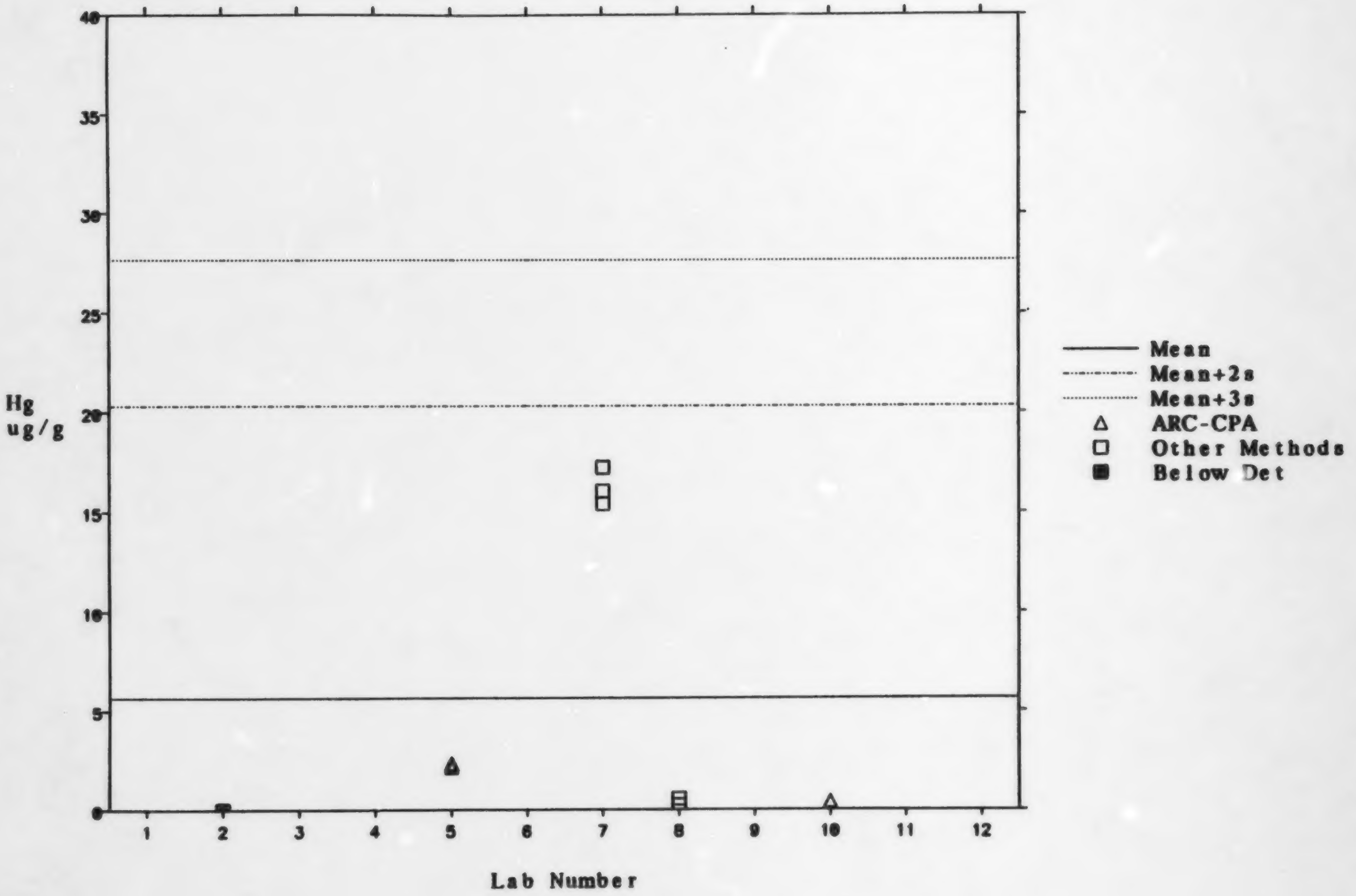


Figure 102. Analysis of Hg in Sludge D

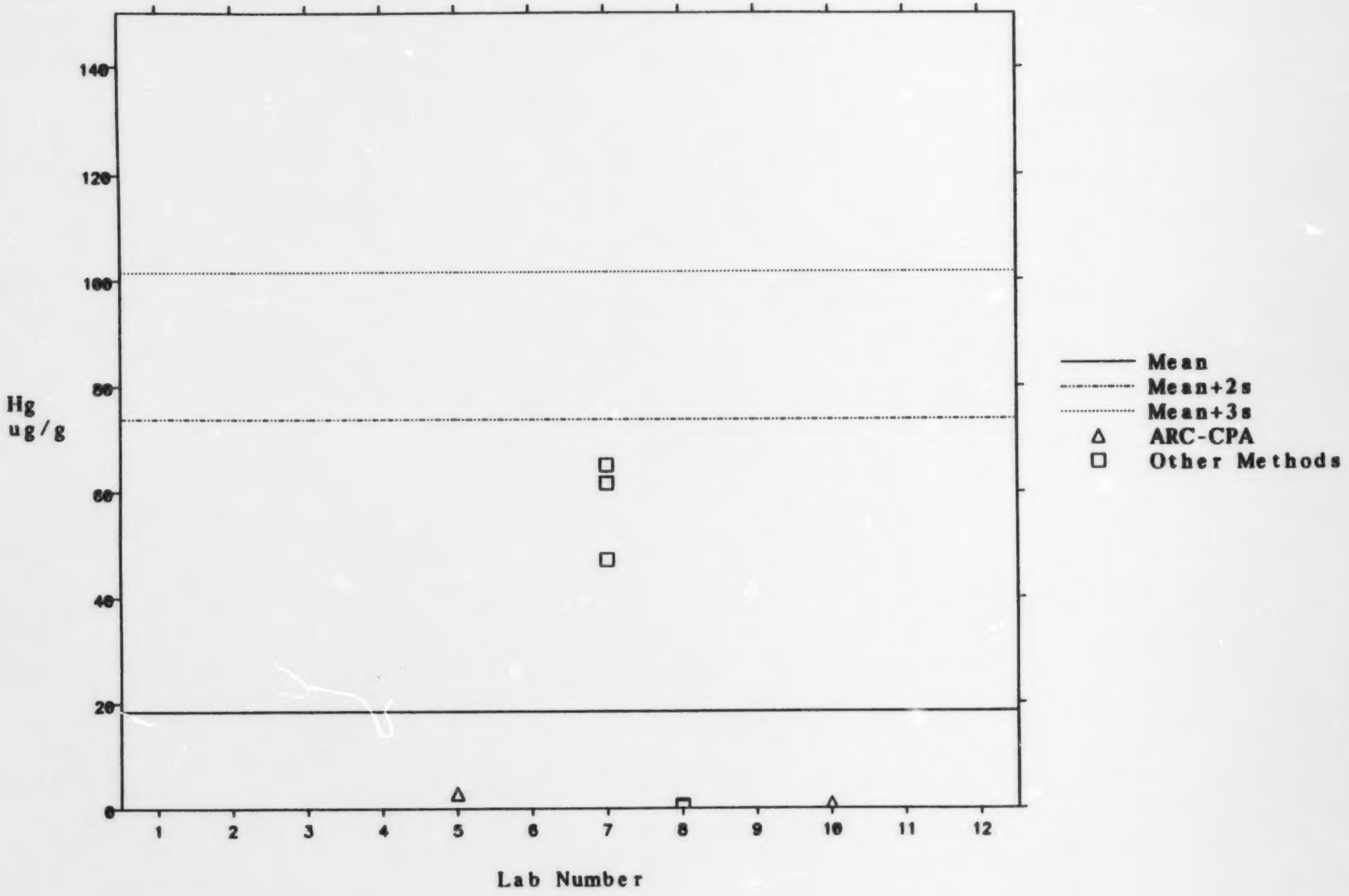


Figure 103. Analysis of Hg in Sludge E

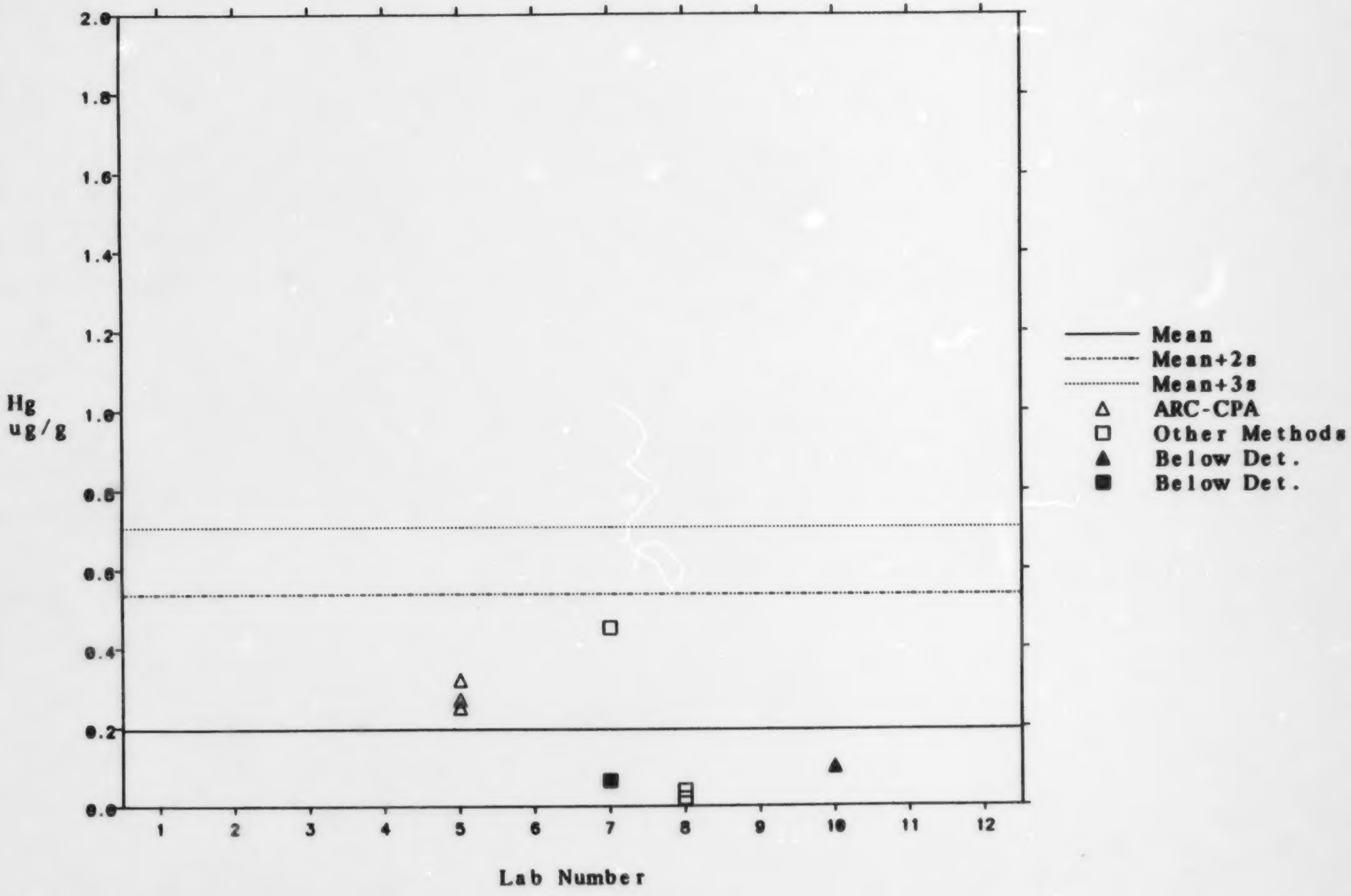


Figure 104. Analysis of Hg in Sludge F

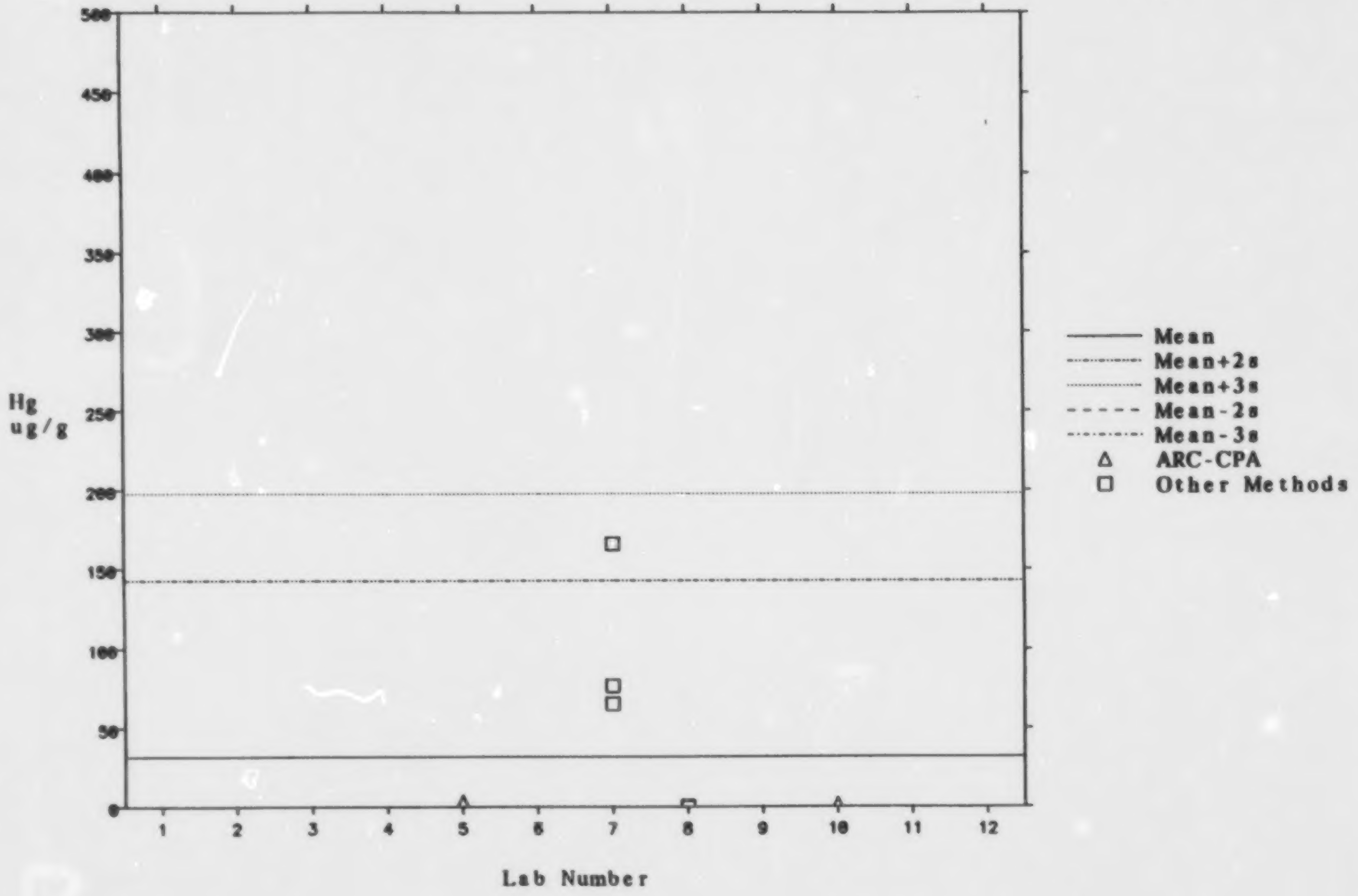


Figure 105. Mean and Confidence Intervals for Mn in an ICP Standard

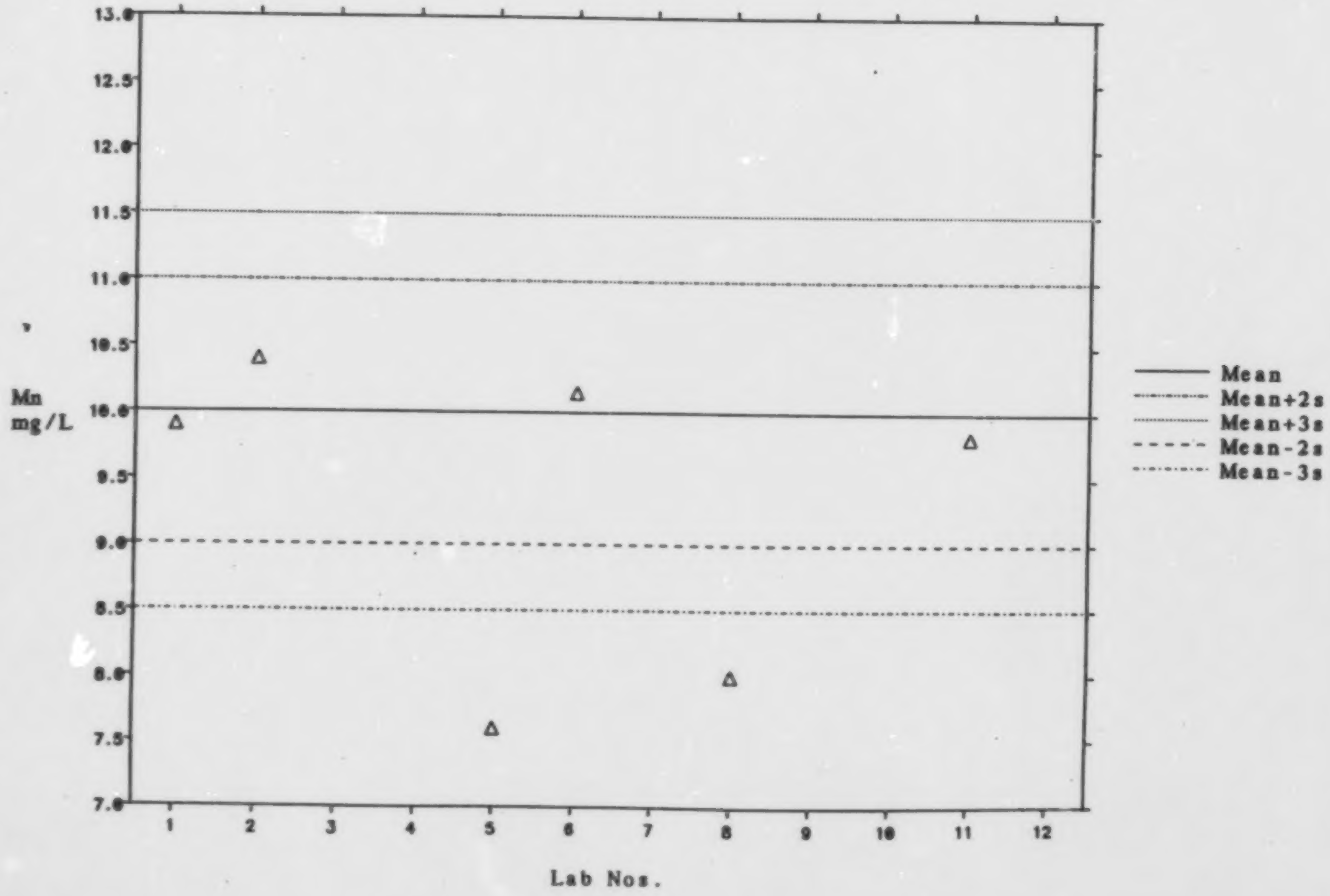


Figure 106. Mean and Confidence Intervals for Mn in Digestate 3

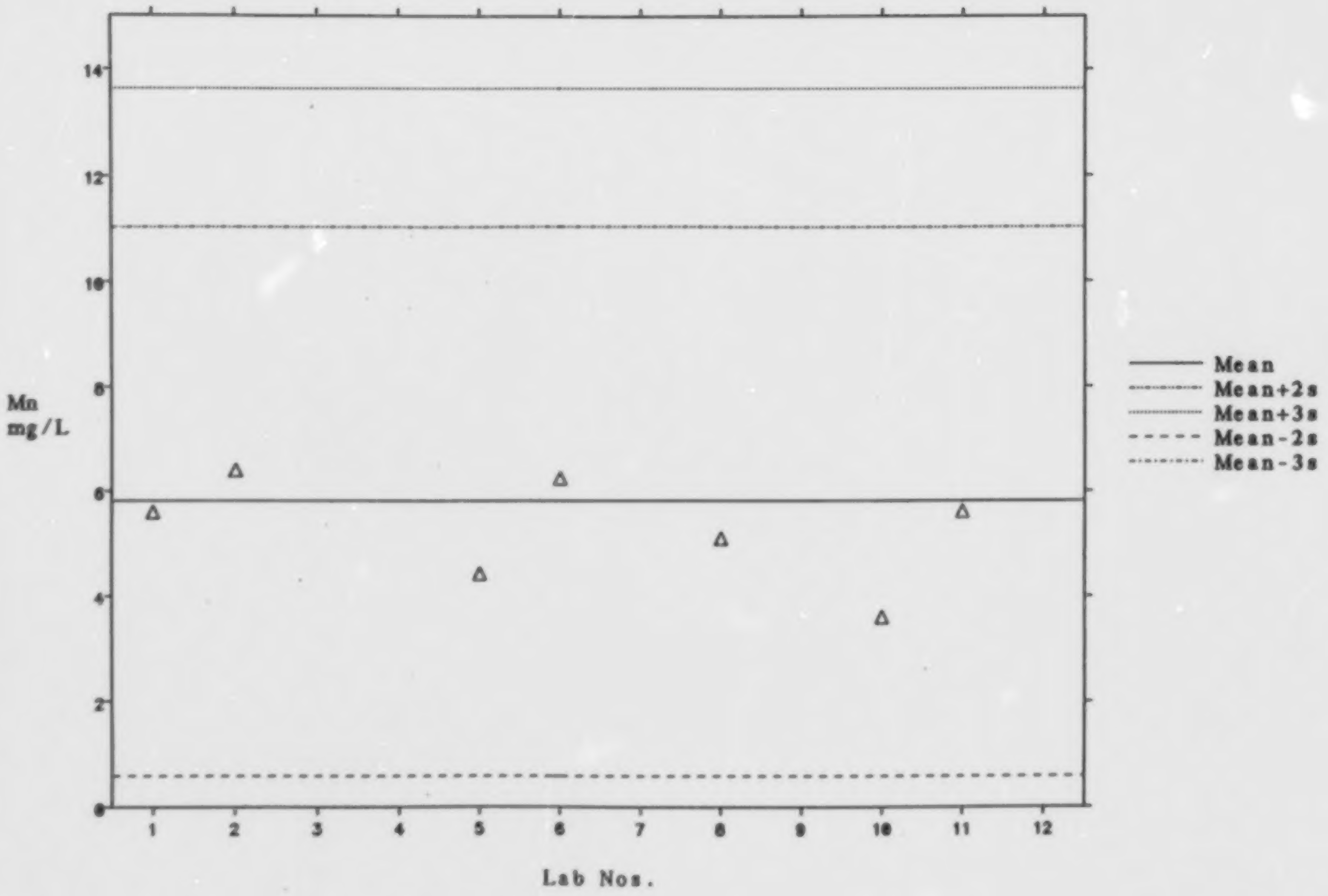
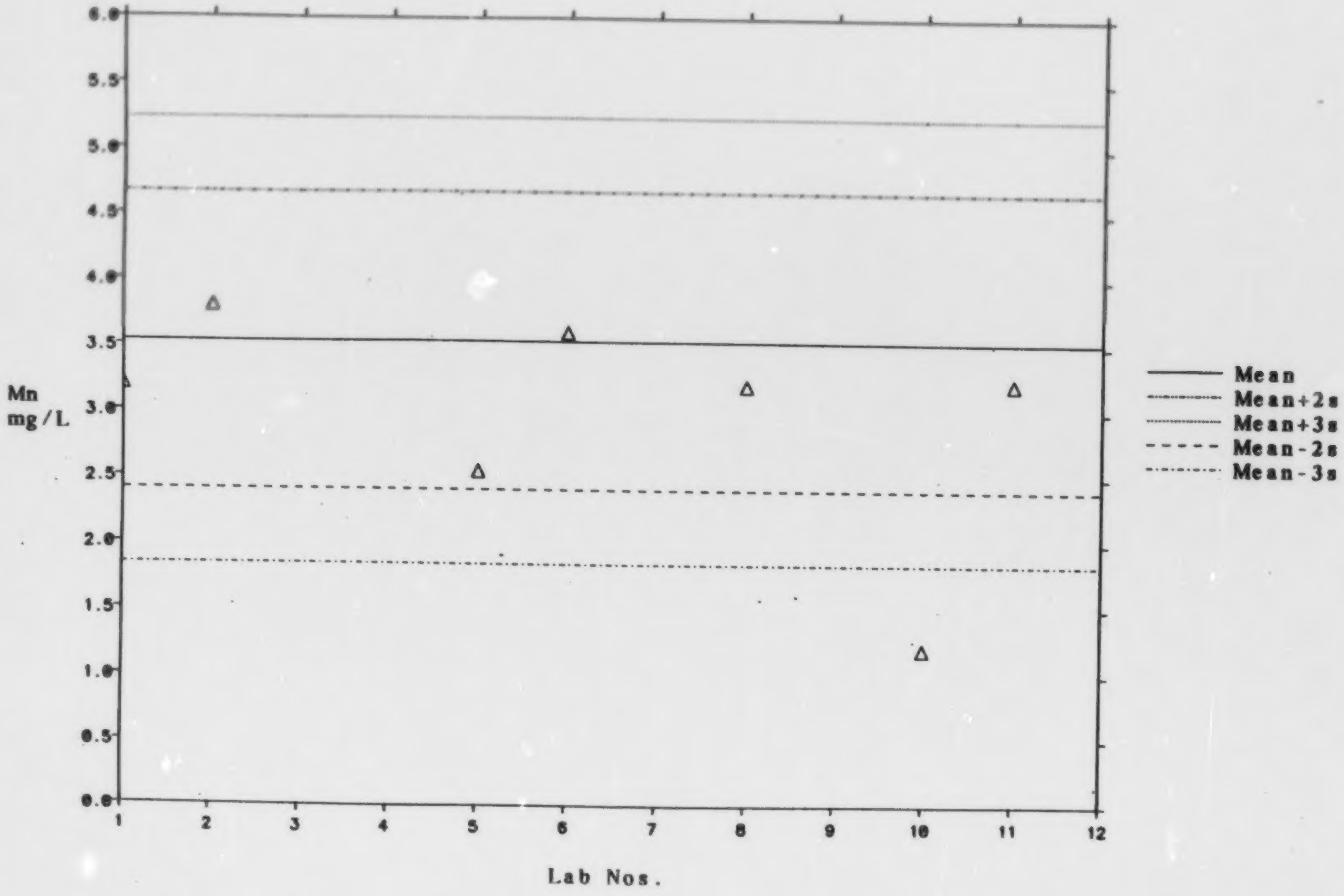


Figure 107. Mean and Confidence Intervals for Mn in Digestate 4



(Figures 106 and 107) were all within the warning control limits except for one lab whose data was lower than the control limit of digestate B. The digestate data shows excellent agreement between the participating labs. Seven labs reported Mn for the standard A and B samples (Figures 108 and 109). Figure 108 shows that all labs reported data within Standards A's warning limit and only two were between the control and warning limits. Standard B values (Figure 109) show that only 3 labs had data within the warning limits, with 4 labs reporting data completely outside the control limits. The discrepancies between the Standard A and B results are likely due to the differences in their control limits. Figures 108 and 109 also show that the results of the ARC-CPA method were not different than those of other methods.

The statistical data for Mn in sludges A to F are shown in Table 12, and their control charts in Figures 110 to 115. Table 12 shows that results (above detection) were reported for all sludges, with number of analyses varying from 31 to 38 samples. The mean Mn values ranged from 7.5 mg/kg for sludge E to 610 for sludge B. The coefficient of variation of the sludges varied from 10 to 36% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 110 to 115 indicate that all the reported Mn data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Mn data shows good agreement between the different labs and for the different methodologies used. The recovery of Mn was very good for all standard samples except Standard B. The variability seems to be dependent on the number of analyses performed and the Mn concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.1.12 Mo

Seven labs reported Mo in the ICP standard (Figure 116), all but one lab were within the control limits. The outlier lab was lower than the lowest control limit. Seven labs reported Mo for the digestate samples

Figure 108. Mean and Confidence Intervals for Mn in Standard A

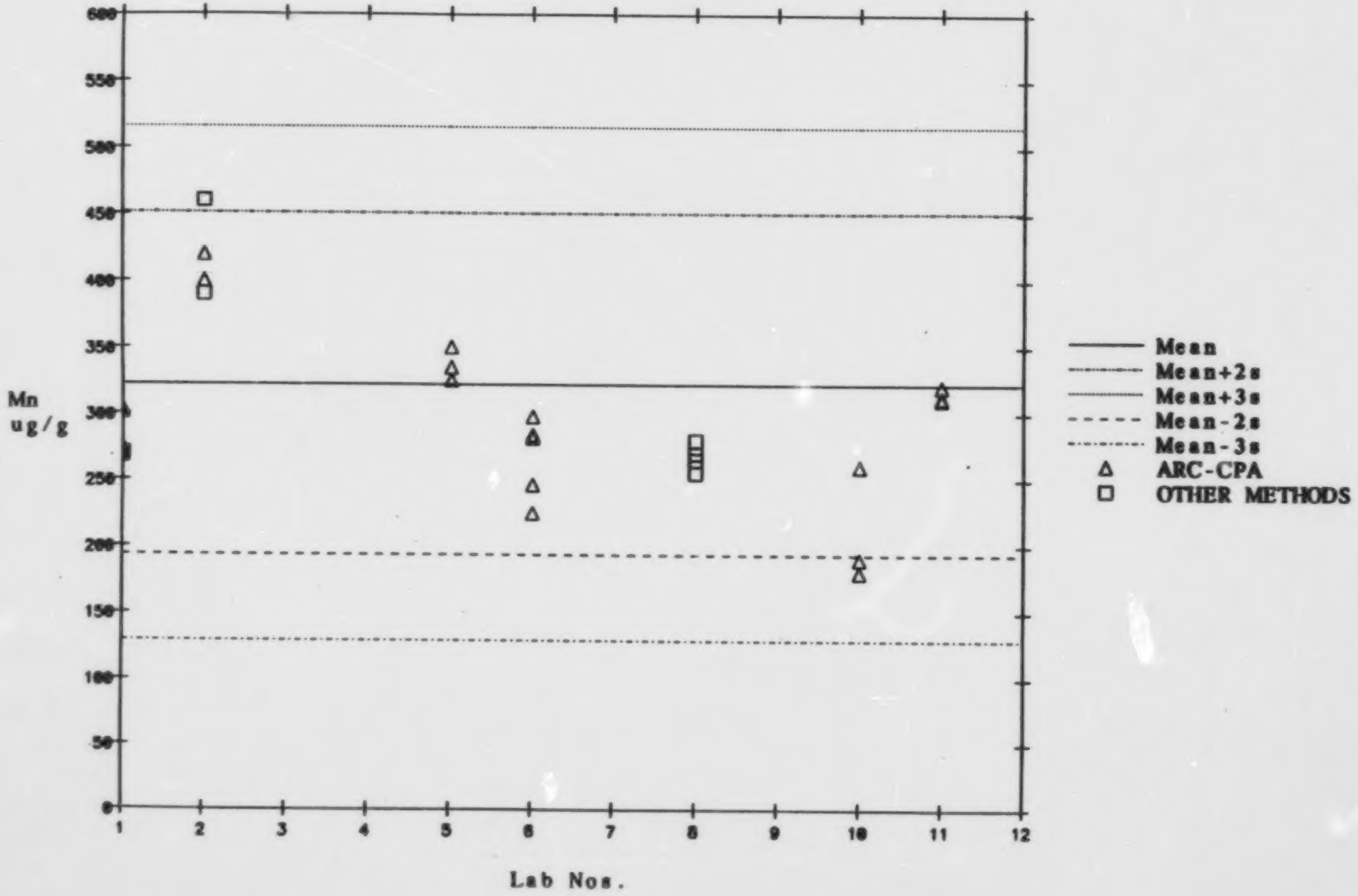


Figure 109. Mean and Confidence Intervals for Mn in Standard B

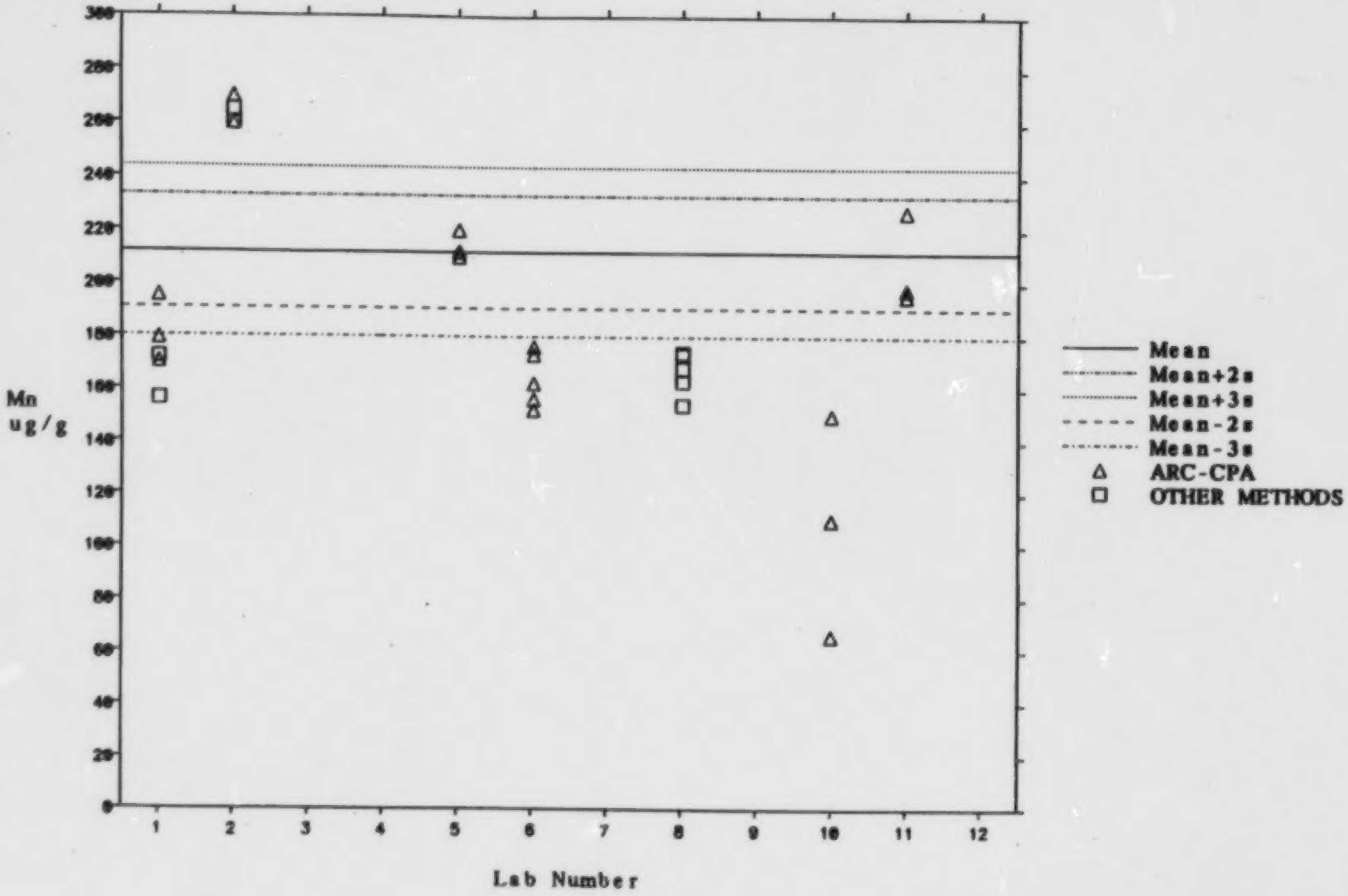


Table 12. Statistical Parameters for Mn (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	33.0	38	32.0	35.0	31.00	31.0
Sum	3050.0	23200	3240.0	3770.0	234.00	6650.0
Mean	92.4	610	101.0	108.0	7.53	214.0
SEM	3.9	10	3.2	6.1	0.37	13.9
Median	89.0	618	101.0	99.0	7.00	190.0
Variance	502.0	3440	327.0	1290.0	4.34	5970.0
StDev	22.4	59	18.1	35.9	2.08	77.3
Maximum	143.0	740	127.0	194.0	16.00	420.0
Minimum	62.0	470	67.0	47.0	4.00	100.0
Range	81.0	270	60.0	147.0	12.00	320.0
Skewness	0.8	-4e-01	-0.4	0.7	2.09	0.9
Kurtosis	0.1	8e-01	-0.3	0.2	8.65	0.3
CV(%)	24.2	10	17.9	33.3	27.70	36.0

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 110. Analysis of Mn in Waste A

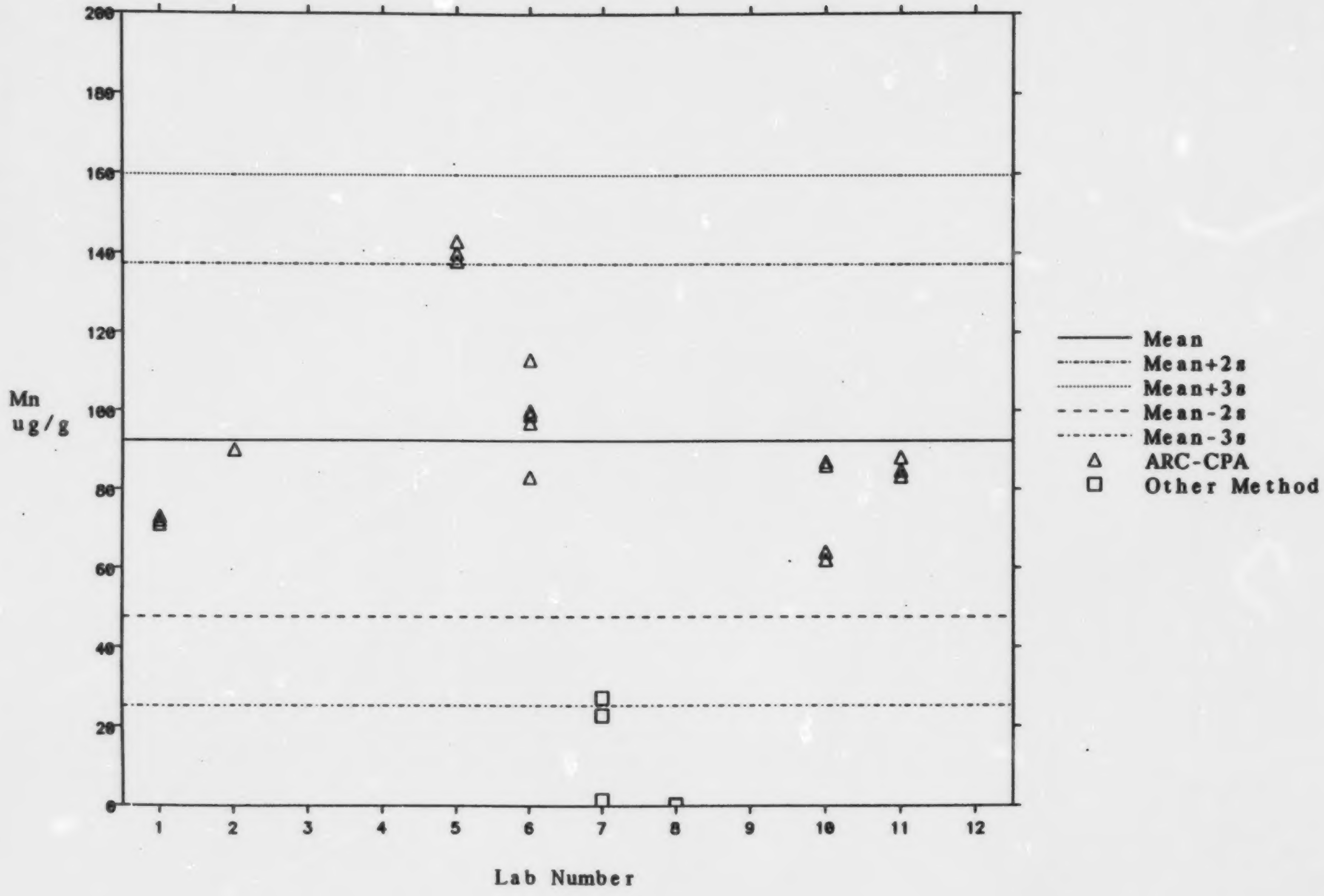


Figure 111. Analysis of Mn in Sludge B

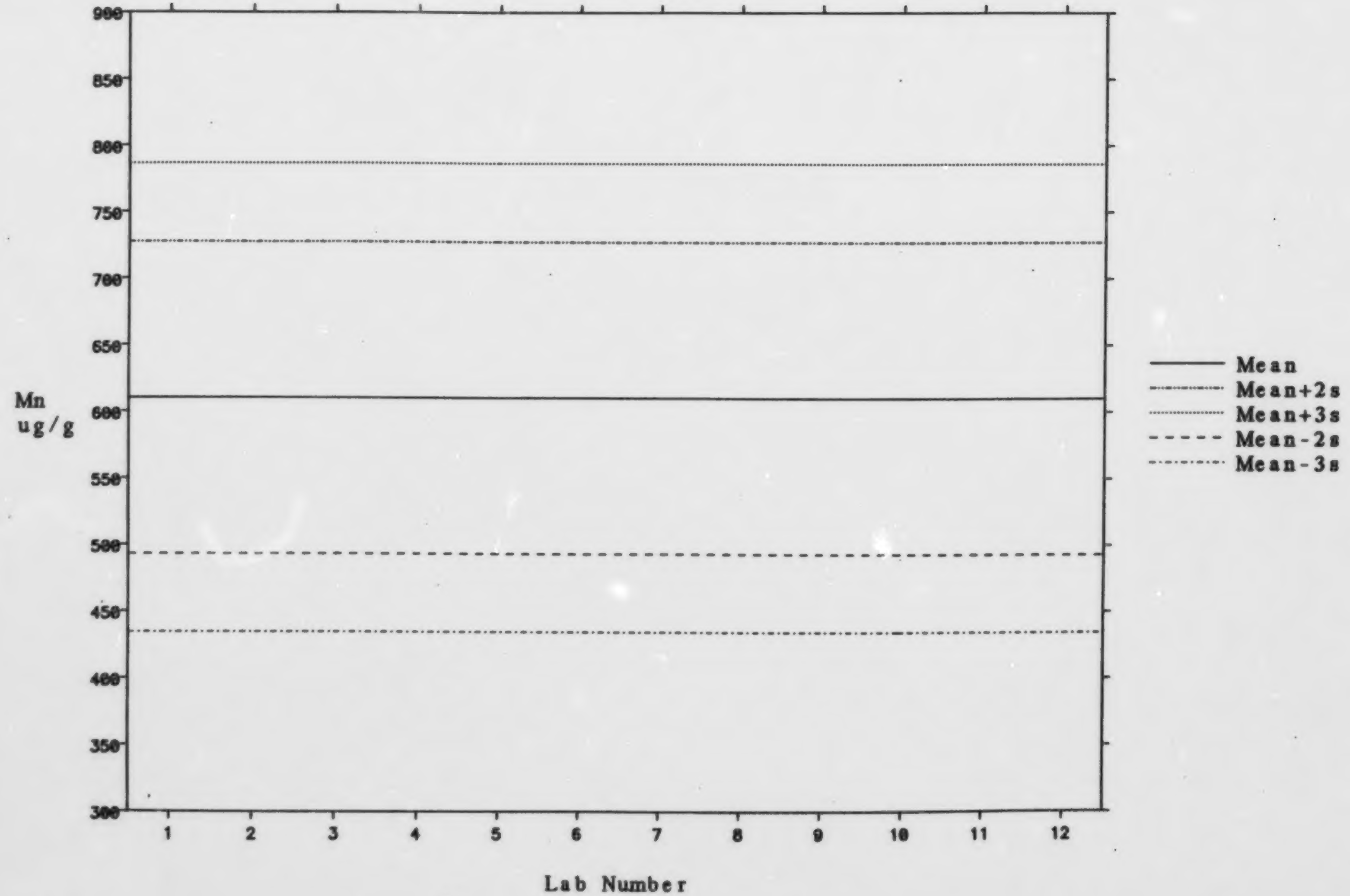
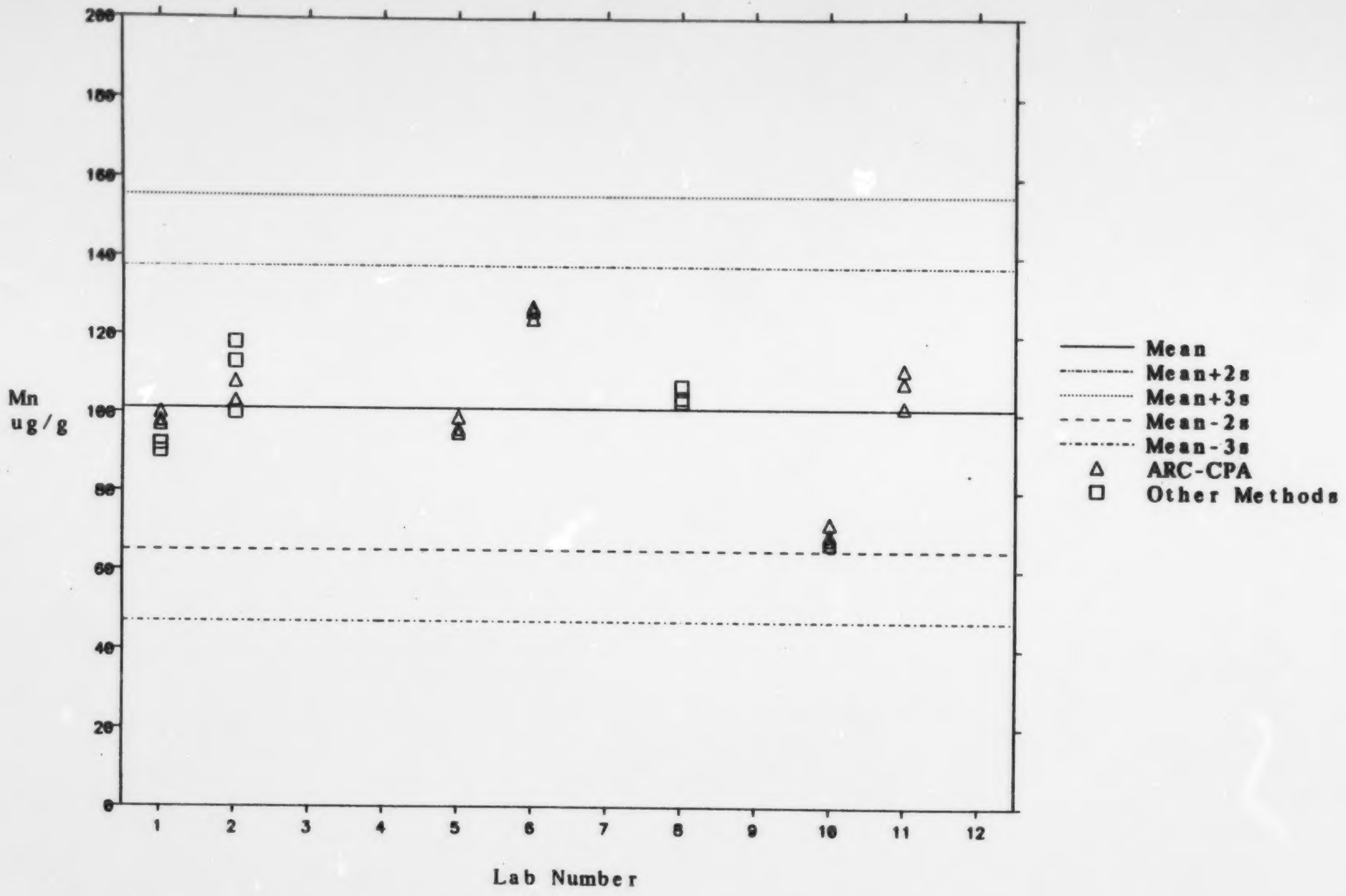


Figure 112. Analysis of Mn in Sludge C



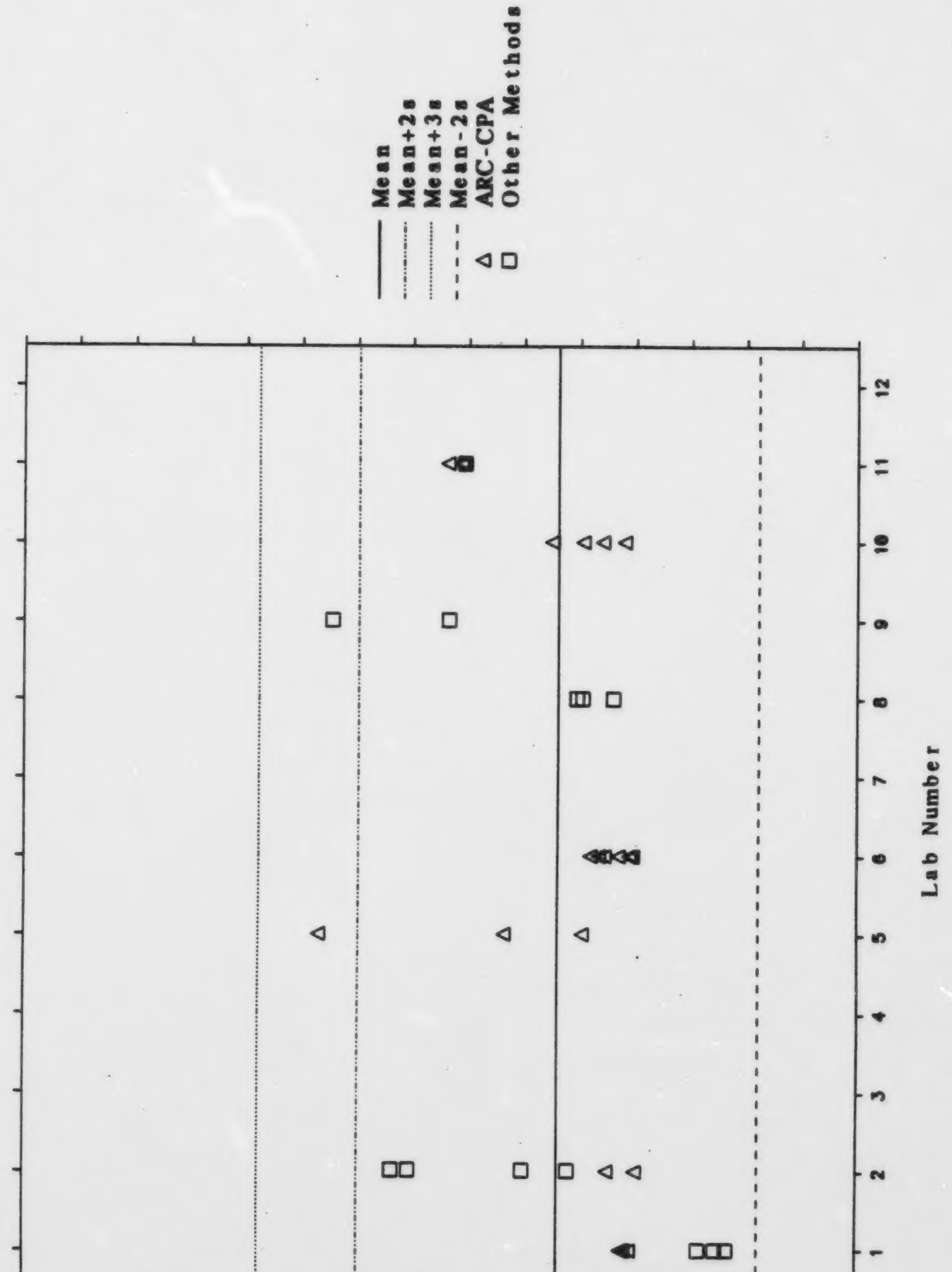


Figure 113. Analysis of Mn in Sludge D

Figure 114. Analysis of Mn in Sludge E

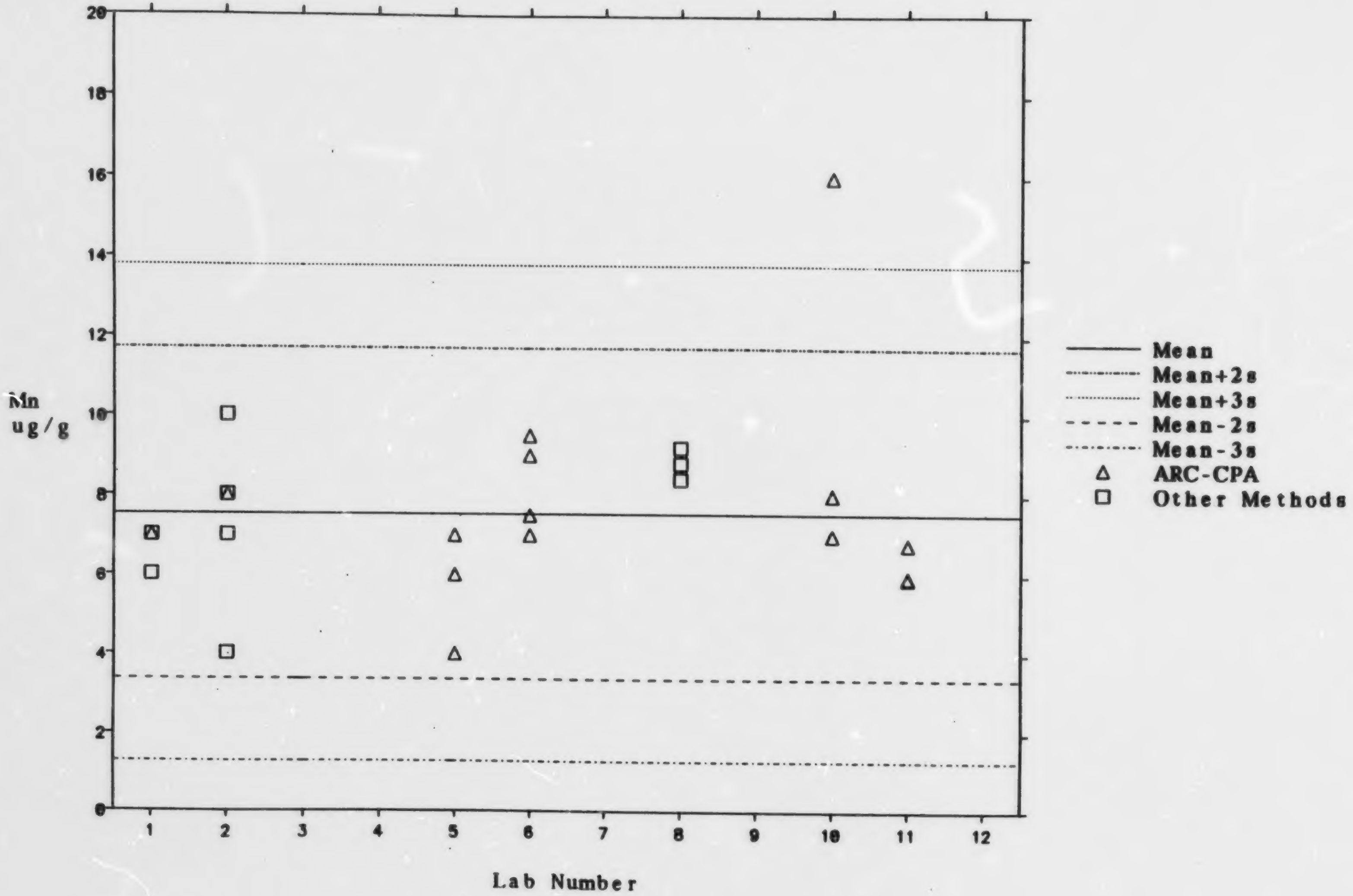


Figure 115. Analysis of Mn in Sludge F

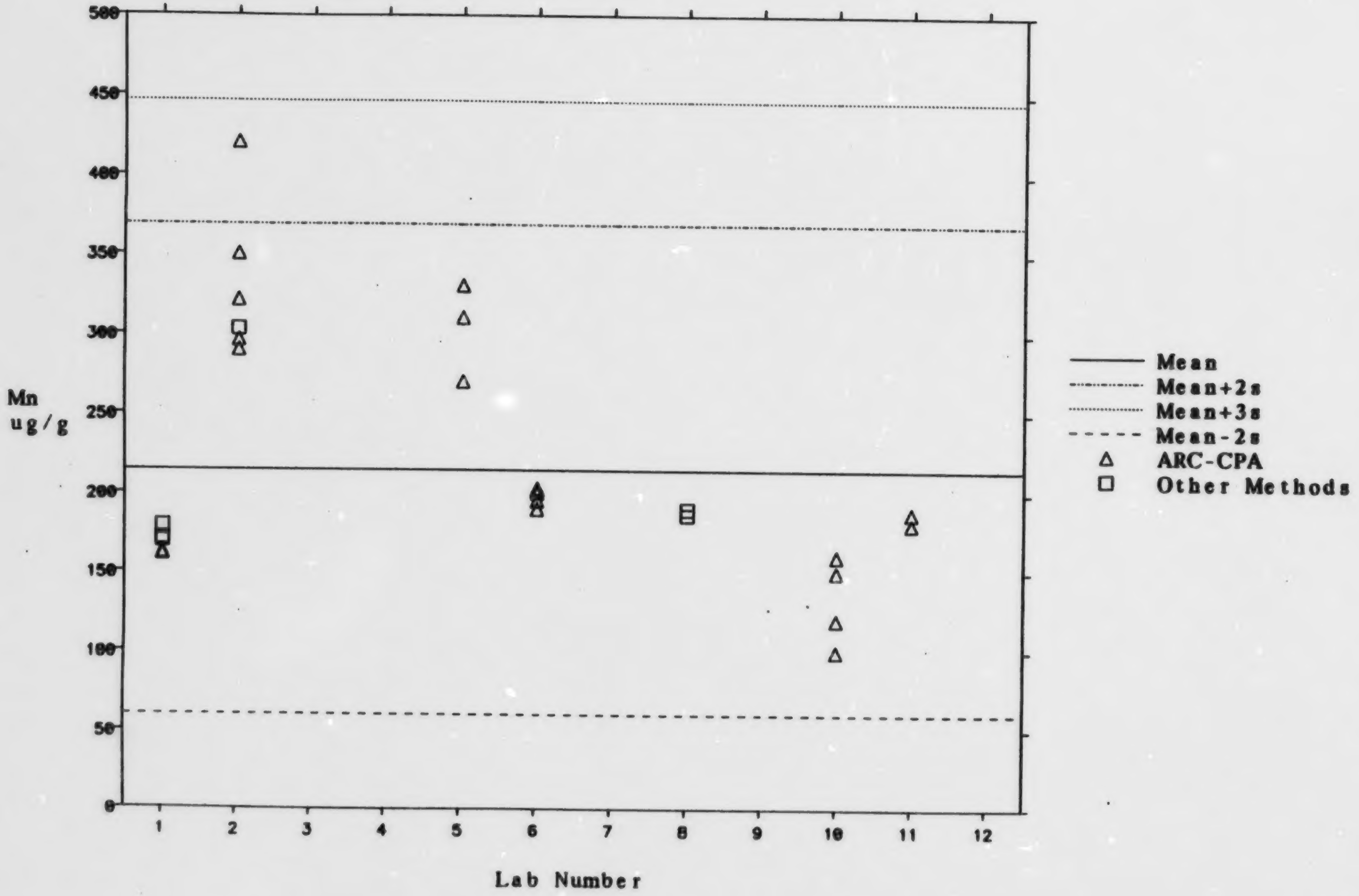
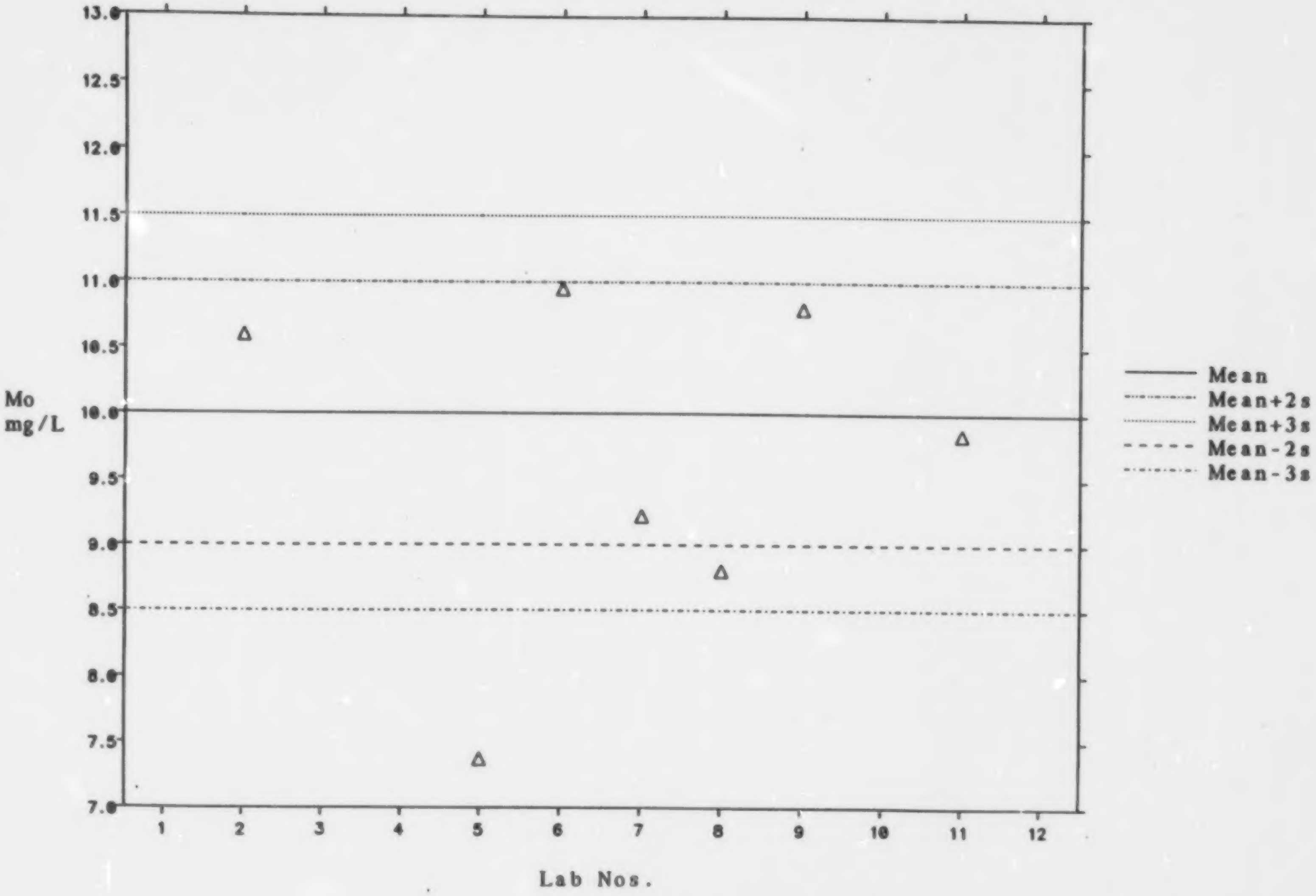


Figure 116. Mean and Confidence Intervals for Mo in an ICP Standard



(Figures 117 and 118). Digestate 3 and 4 data (Figures 117 and 118) were all within the warning limits, indicating close agreement between the labs and the actual digestates' Mo concentrations. Five labs reported Mo for the standard A and seven labs for the standard B samples (Figures 119 and 120). Figure 119 shows that all labs reported data within and outside Standards A's control limits. Standard B values (Figure 120) show that 4 labs had data within the warning limits, and 3 labs reporting data completely outside the control limits. Figure 119 also shows that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Mo in sludges A to F are shown in Table 13, and their control charts in Figures 121 to 126. Table 13 shows that results (above detection) were reported for all sludges, with number of analyses varying from 17 to 24 samples. The mean Mo values ranged from 8.9 mg/kg for sludge D to 24.5 for sludge C. The coefficient of variation of the sludges varied from 45 to 176%. The CV values did not seem to be related to the number of reported results or the measured Mo concentrations.

Figures 121 to 126 indicate that all the reported Mo data (except sludge A) fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Mo data for the standards shows good recovery and agreement between the different labs and for the different methodologies used. The variability did not seem to be dependent on the number of analyses performed or the Mo concentration.

4.1.1.13 Ni

Seven labs reported Ni in the ICP standard (Figure 127), all but two labs were within the control limits. The two outlier labs were both lower than the control limit. Eight labs reported Ni for the digestate

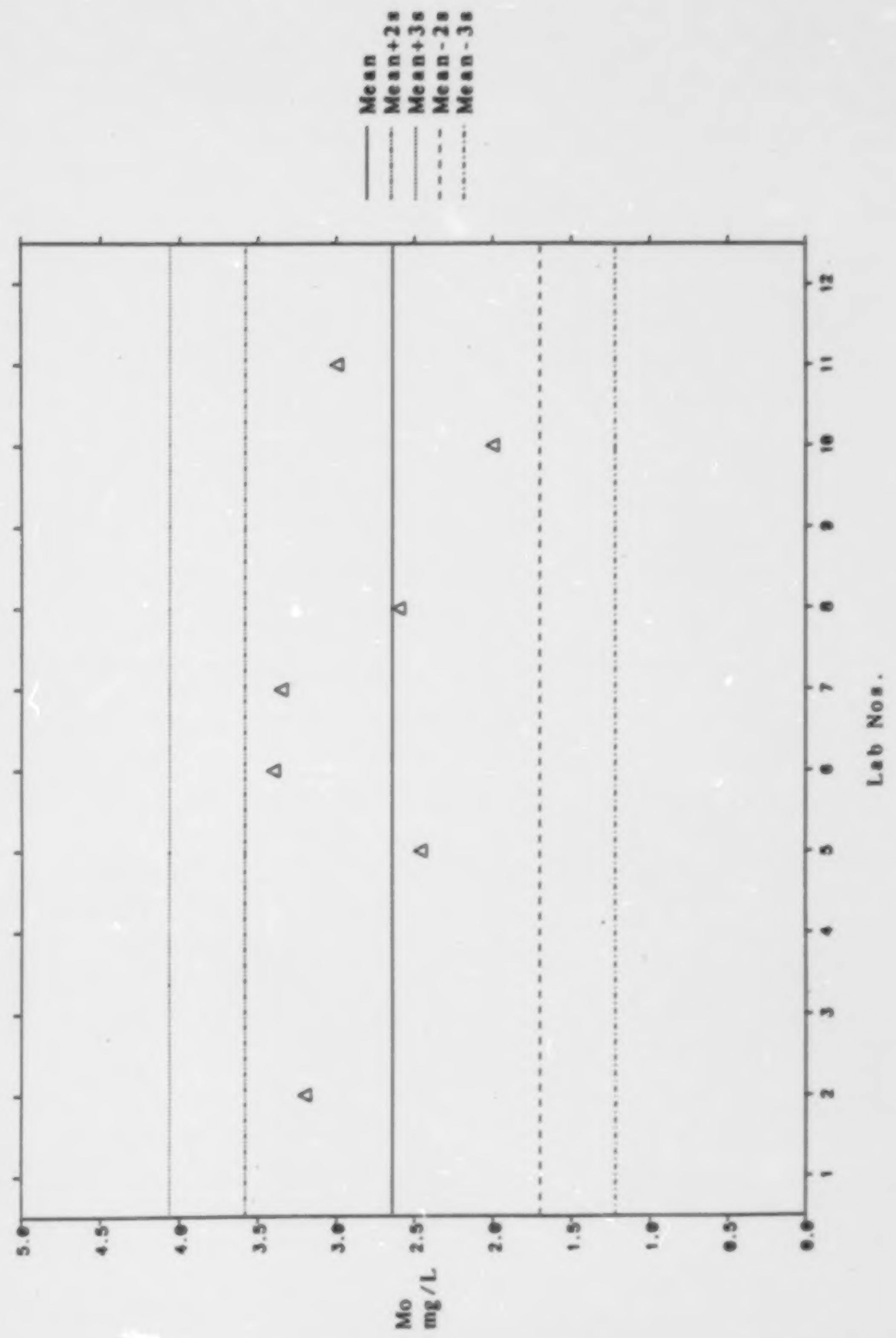


Figure 117. Mean and Confidence Intervals for Mo in Digestate 3

Figure 118. Mean and Confidence Intervals for Mo in Digestate 4

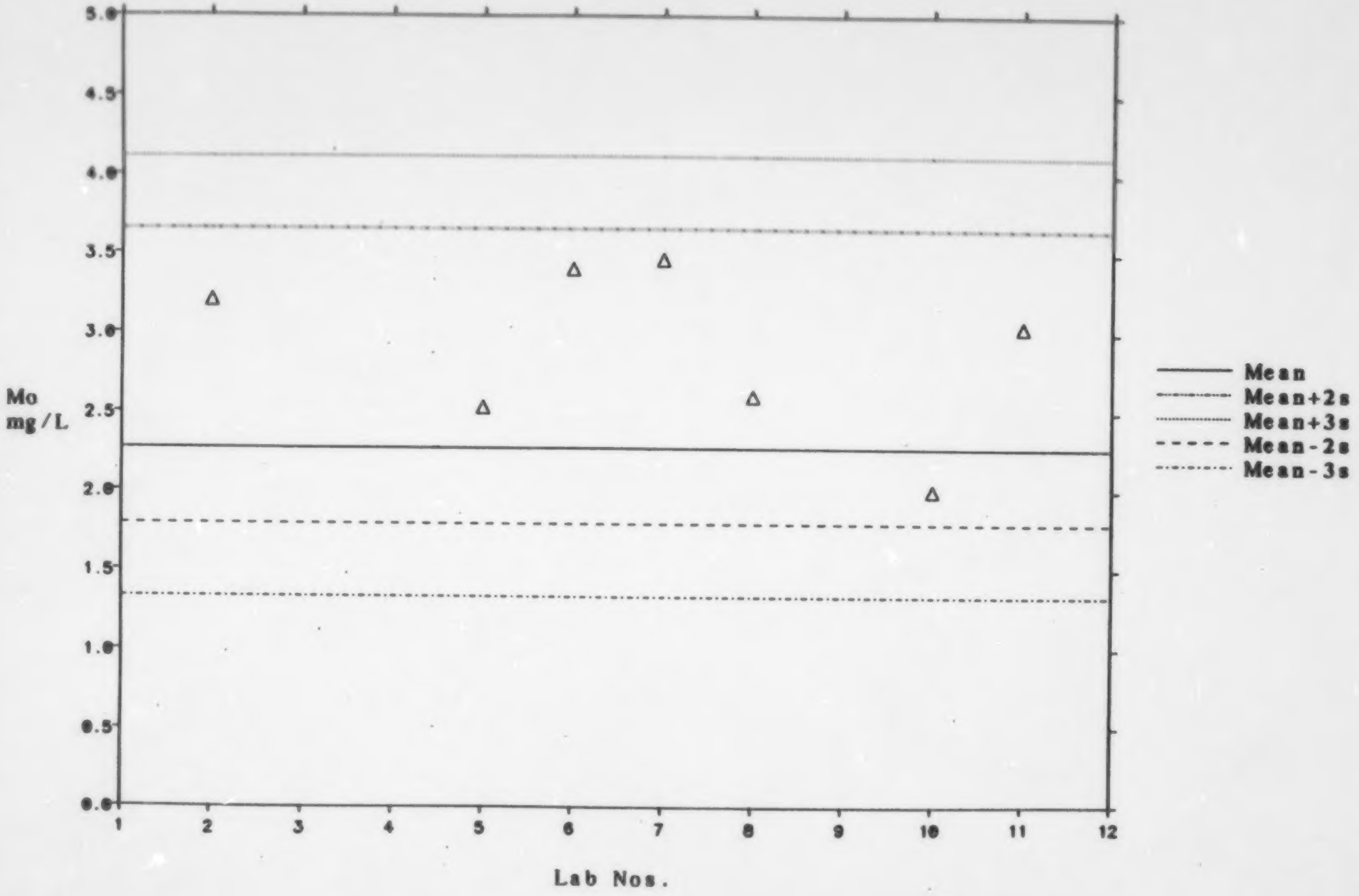


Figure 119. Mean and Confidence Intervals for Mo in Standard A

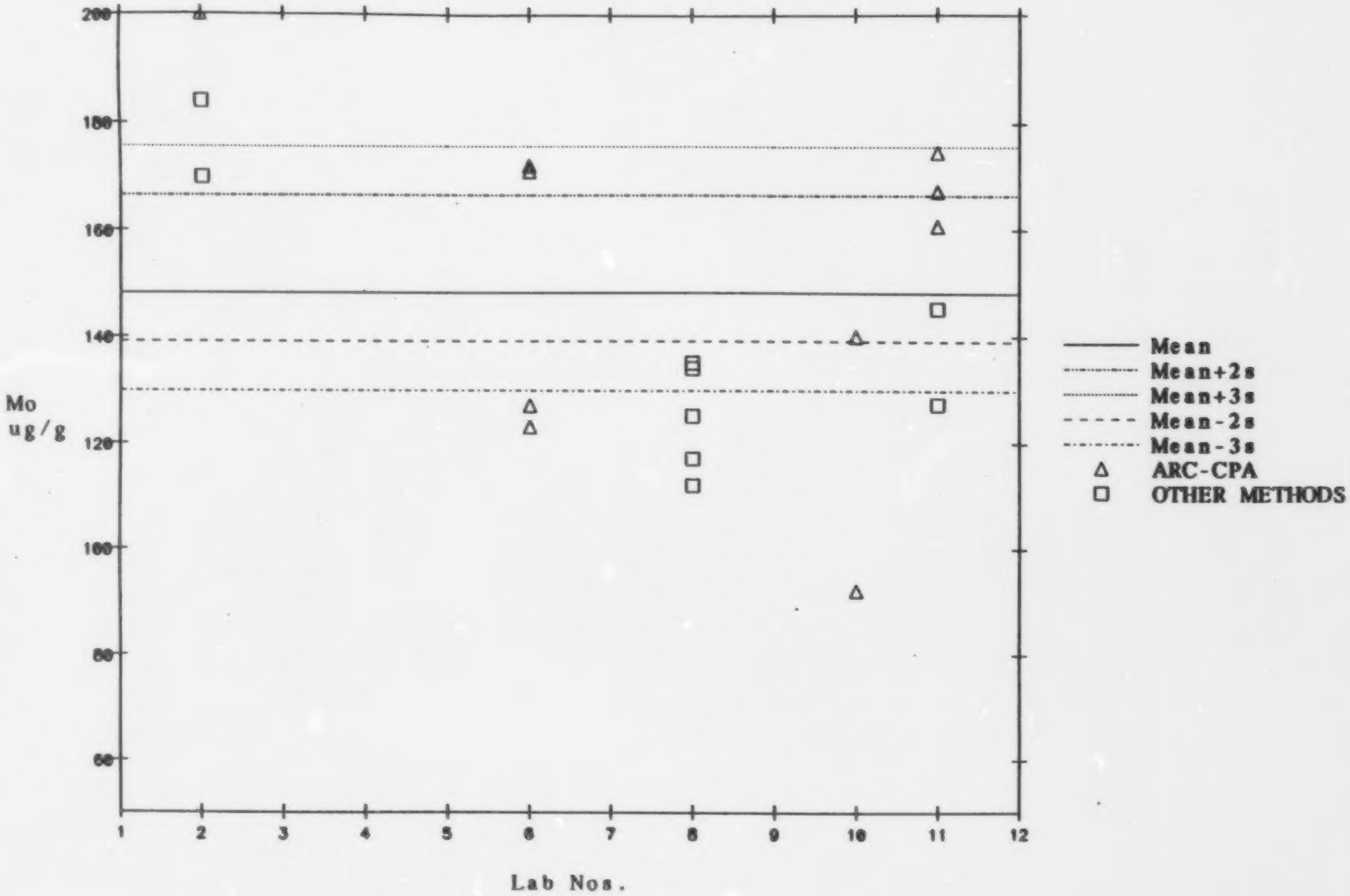


Figure 120. Mean and Confidence Intervals for Mo in Standard B

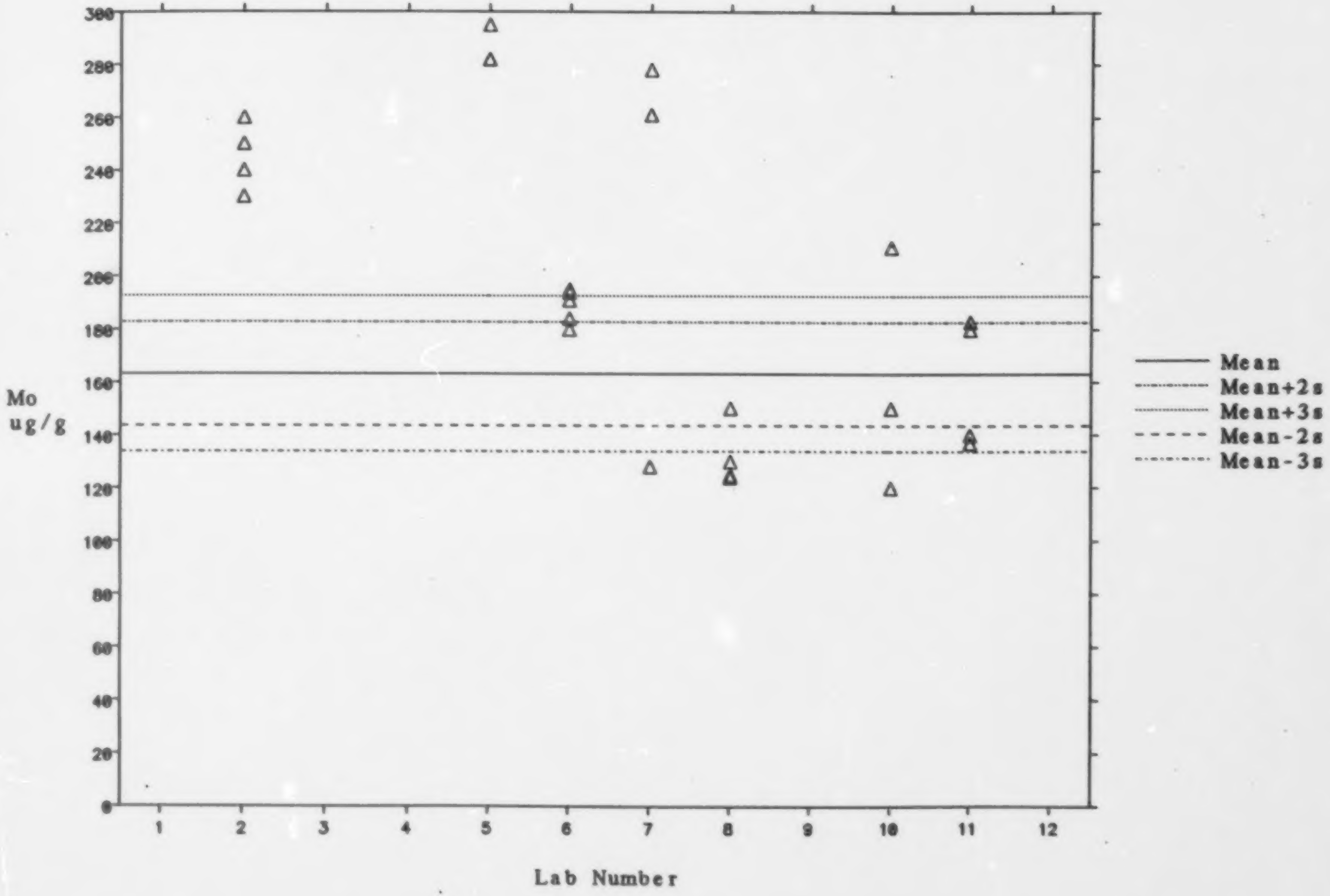


Table 13. Statistical Parameters for Mo (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	24.00	21.00	24.0	23.00	17.00	20.00
Sum	307.00	452.00	589.0	206.00	272.00	401.00
Mean	12.80	21.50	24.5	8.94	16.00	20.00
SEM	3.79	5.06	8.8	0.85	1.97	5.76
Median	7.00	19.00	10.0	7.00	12.90	10.90
Variance	345.0	539.00	1870.0	16.50	65.60	663.00
StDev	18.60	23.20	43.2	4.06	8.10	25.70
Maximum	79.50	79.90	138.0	19.70	35.00	86.80
Minimum	3.00	1.98	5.0	5.00	8.26	4.00
Range	76.50	77.90	133.0	14.70	26.70	82.80
Skewness	3.13	1.69	2.4	1.50	1.55	2.06
Kurtosis	9.21	2.01	4.2	1.41	1.31	2.83
CV(%)	145.00	108.00	176.0	45.40	50.70	128.00

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 121. Analysis of Mo in Waste A

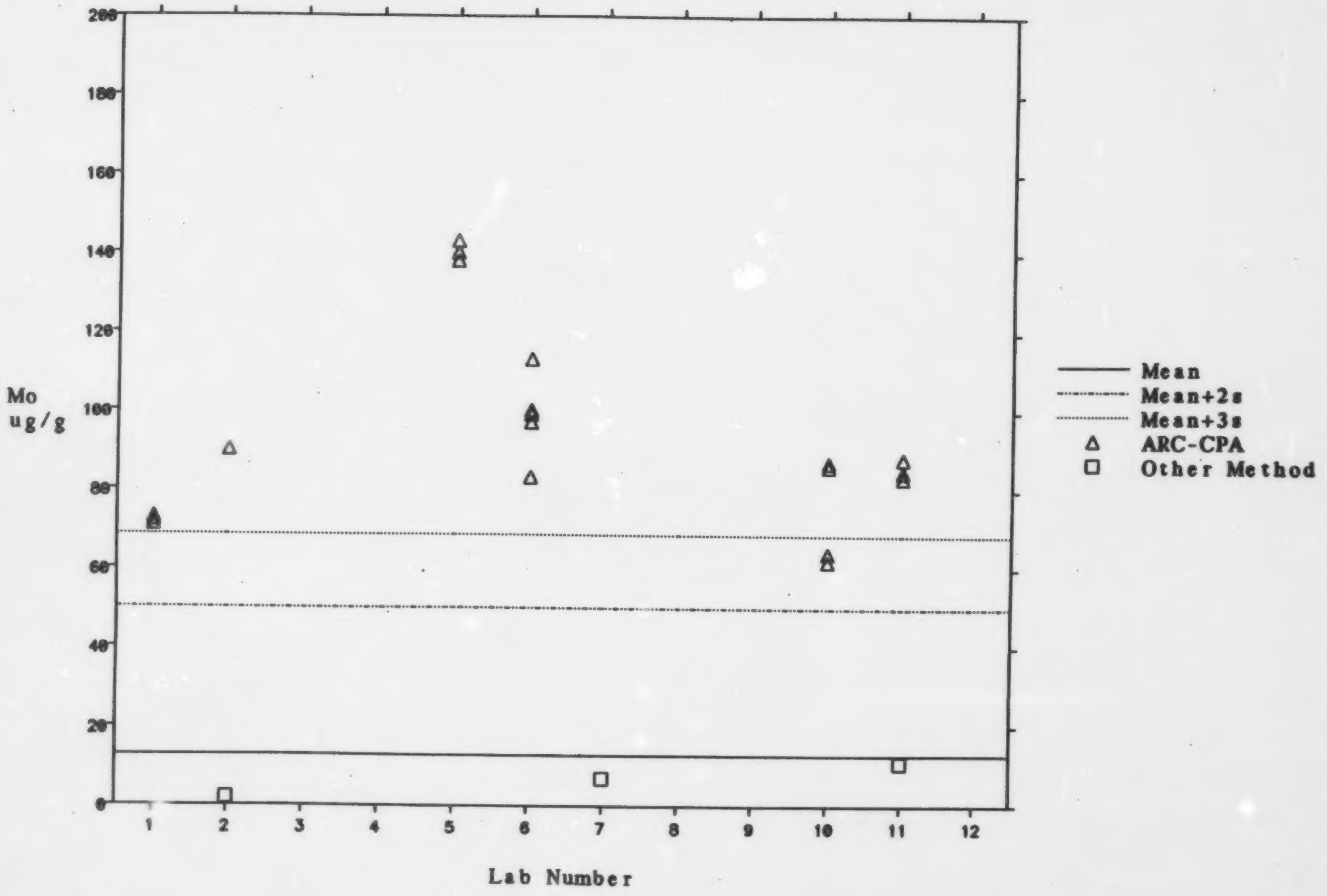


Figure 122. Analysis of Mo in Sludge B

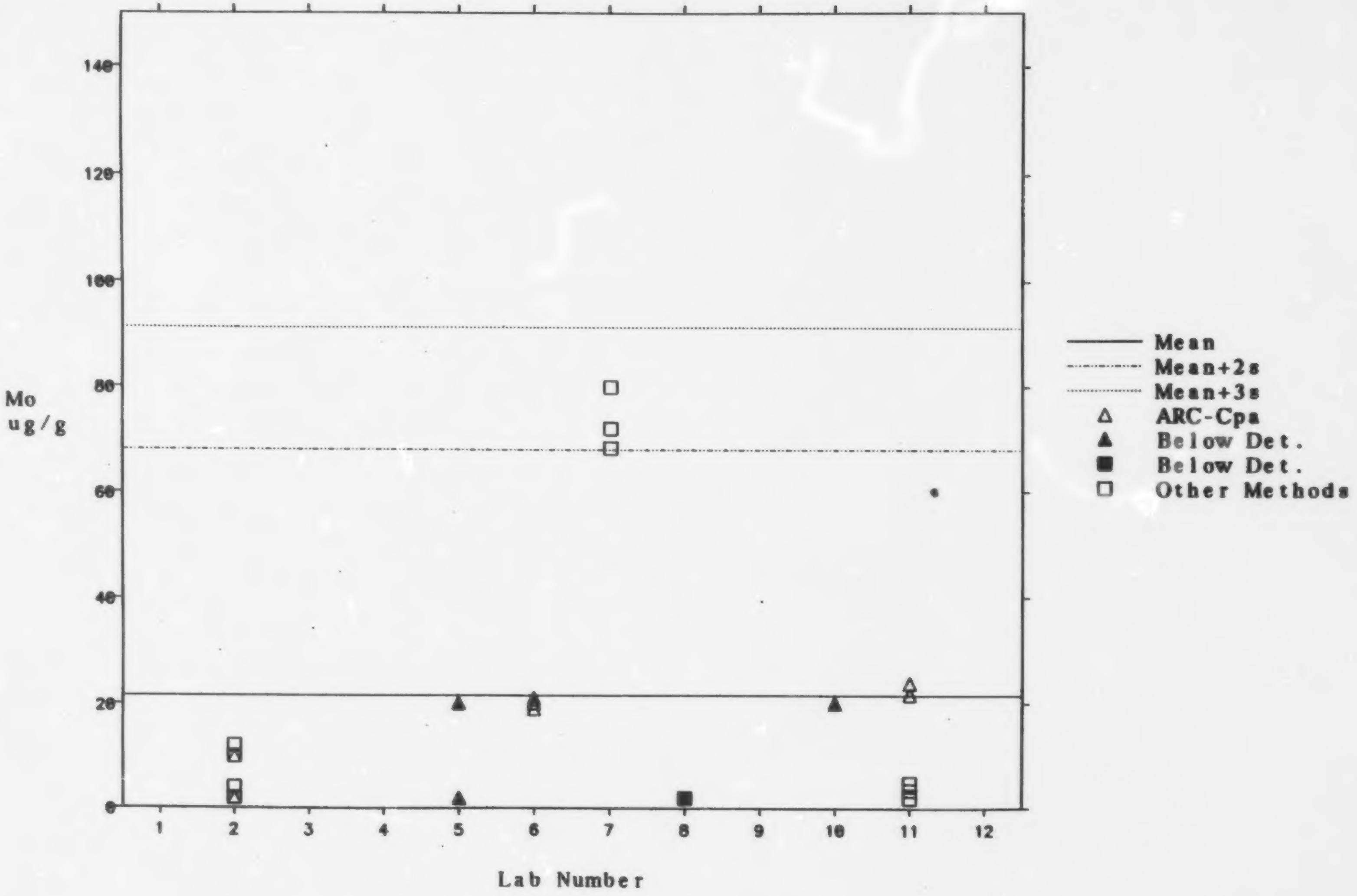


Figure 123. Analysis of Mo in Sludge C

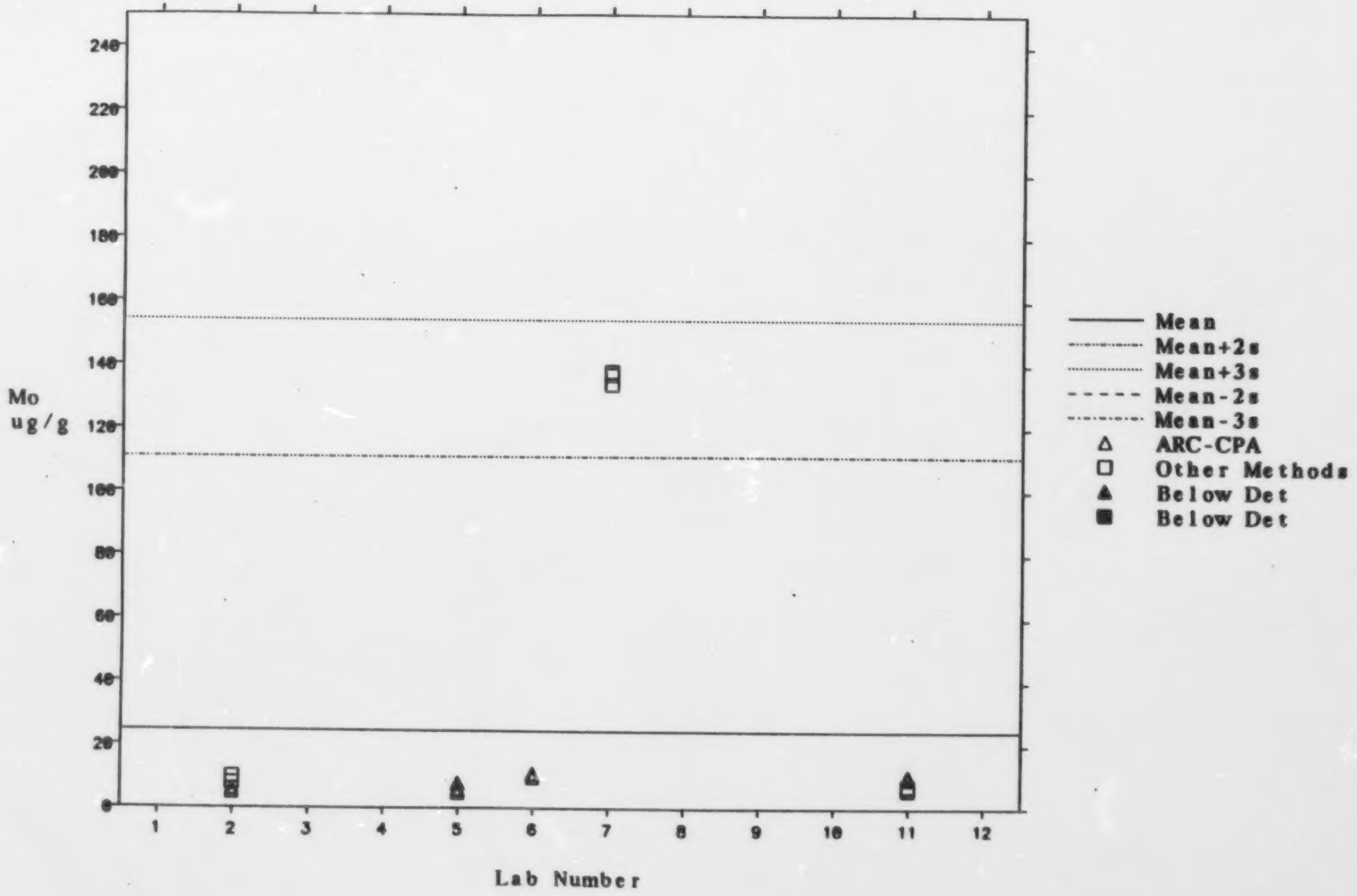


Figure 124. Analysis of Mo in Sludge D

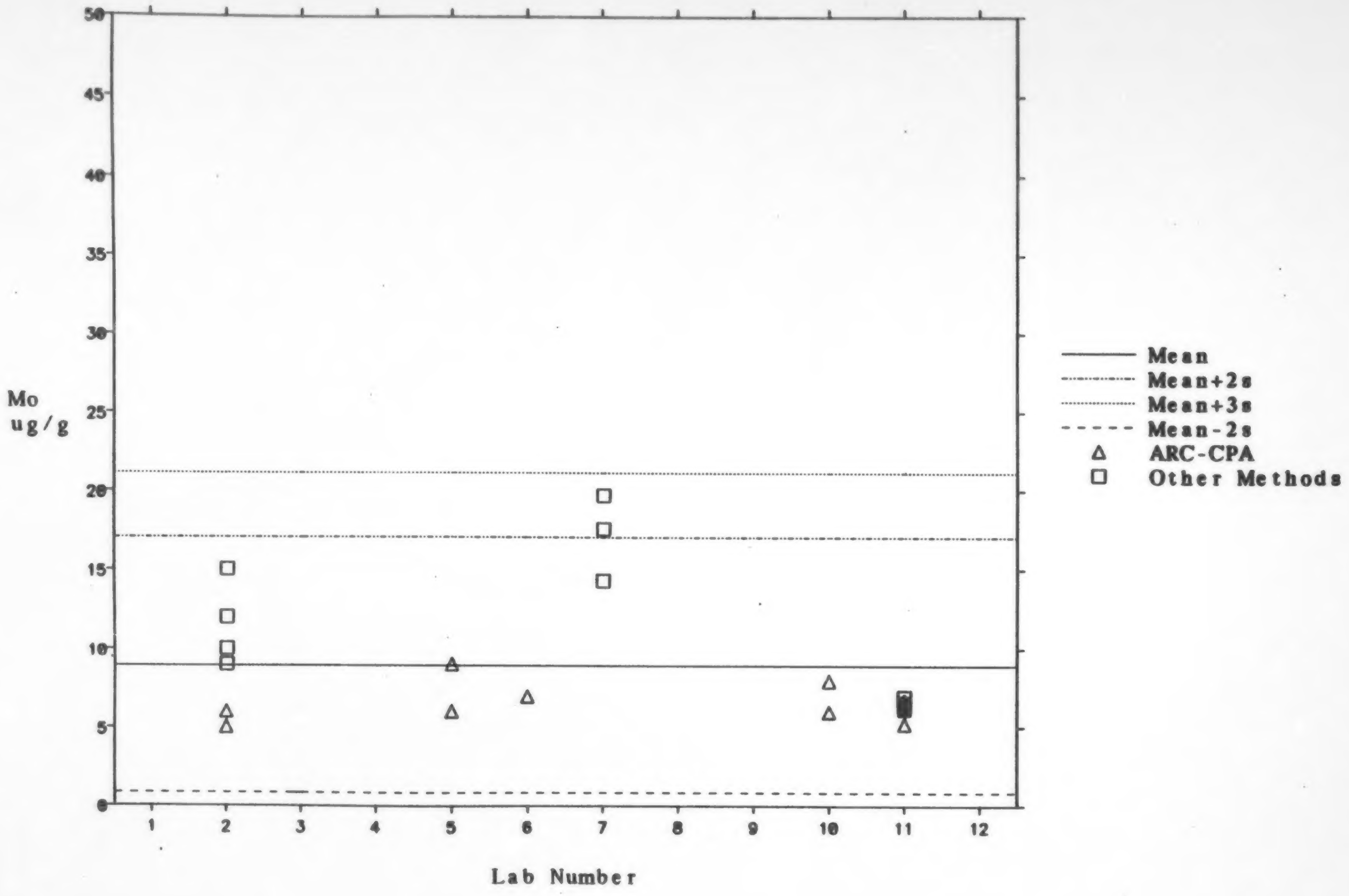


Figure 125. Analysis of Mo in Sludge E

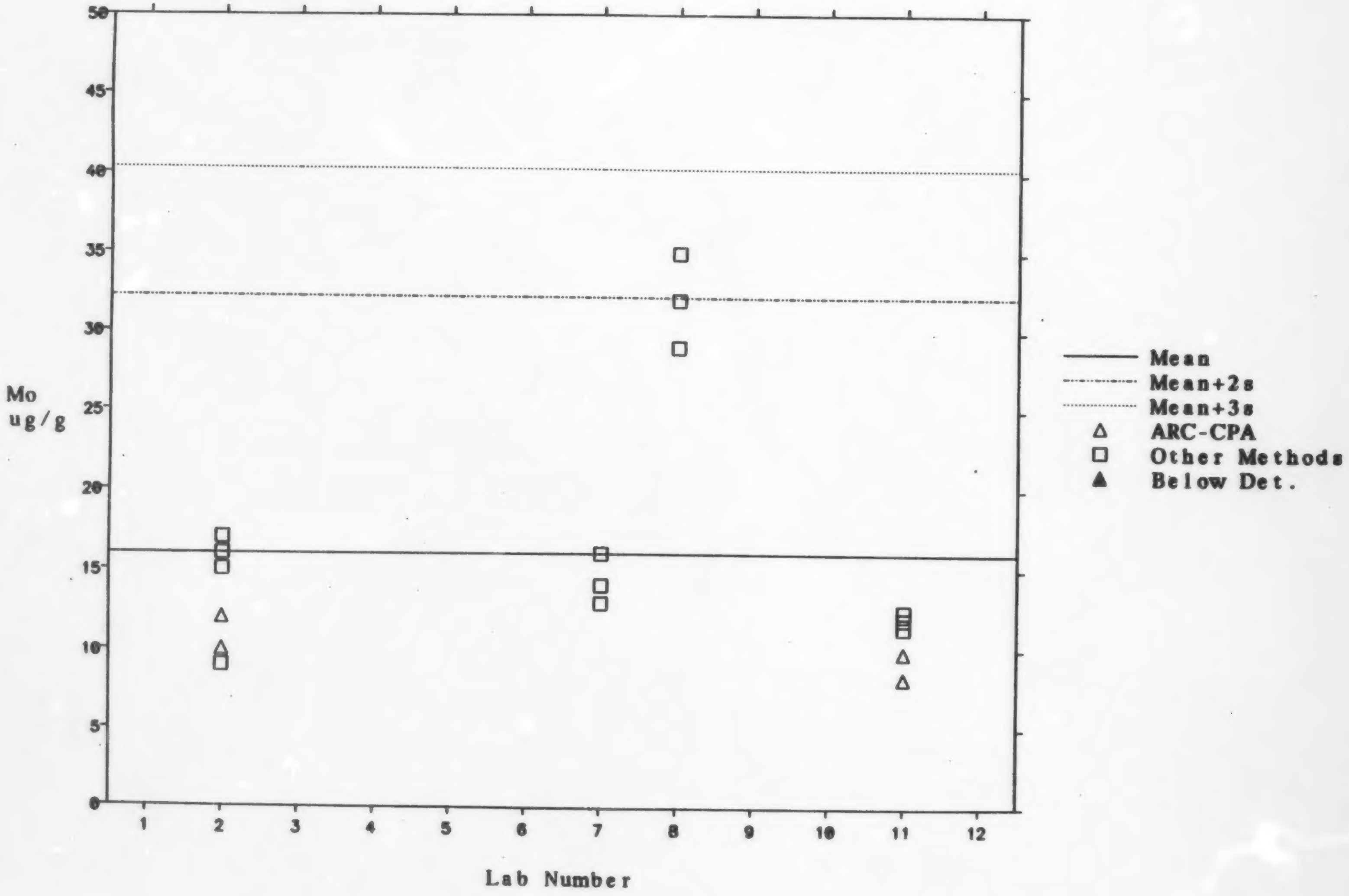


Figure 126. Analysis of Mo in Sludge F

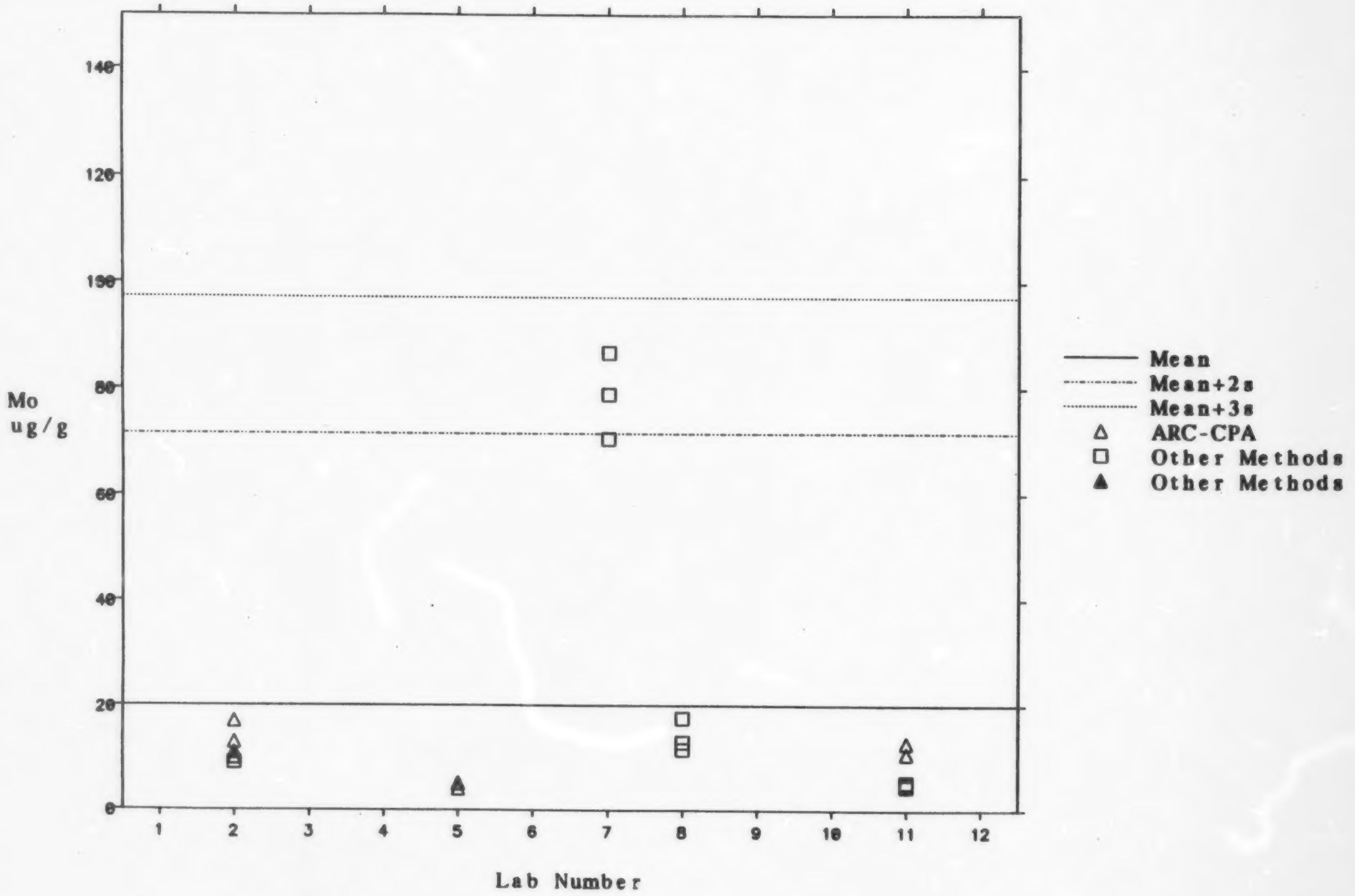
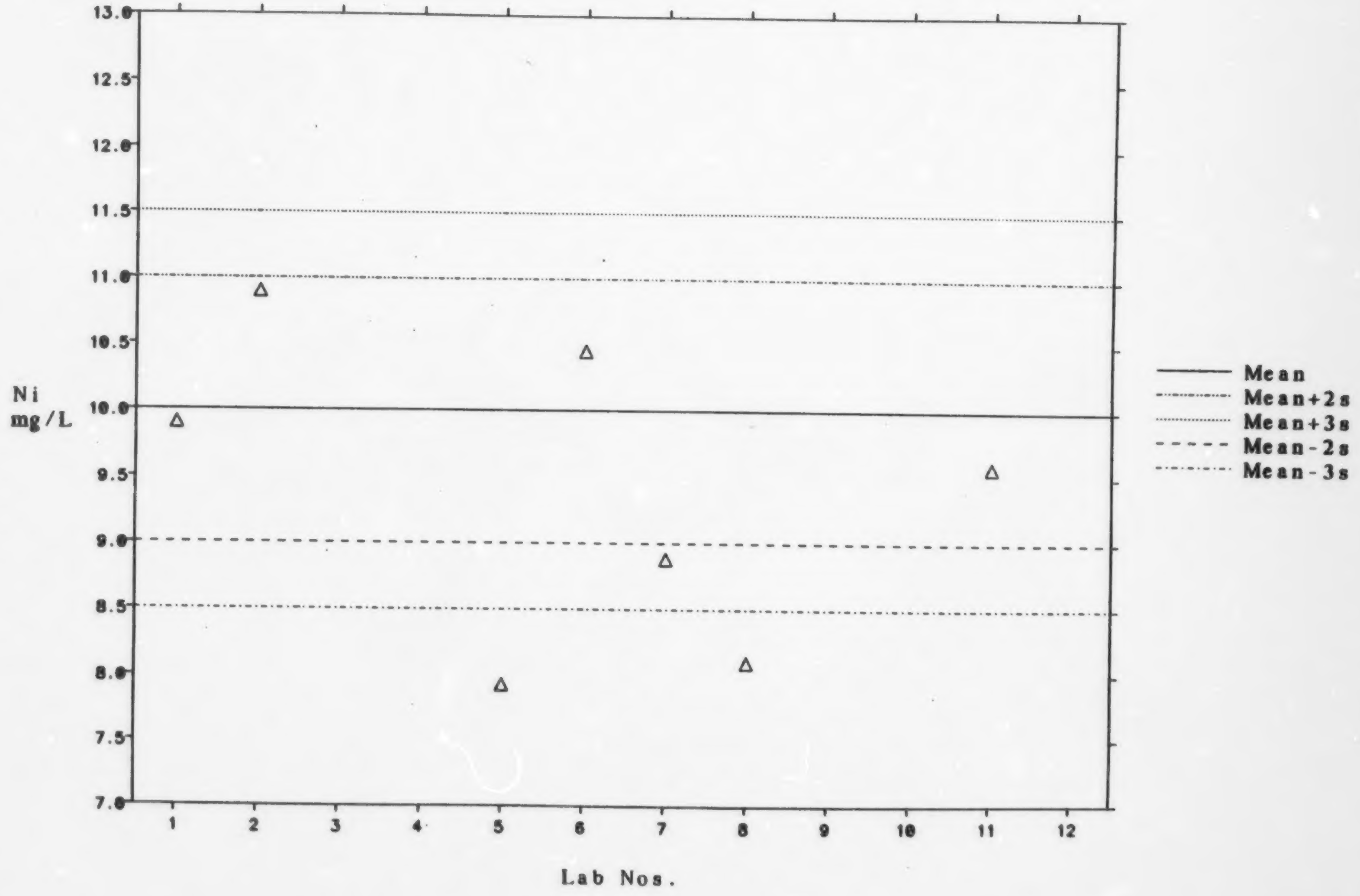


Figure 127. Mean and Confidence Intervals for Ni in an ICP Standard



samples (Figures 128 and 129). Digestate 3 and 4 data (Figures 128 and 129) were all within the warning limits, showing good recovery and agreement between all labs. Eight labs reported Ni for the standard A and B samples (Figures 130 and 131). Figure 130 shows that 3 labs reported data completely outside Standards A's control limits, 2 labs were both inside and outside the limits, and the other 2 were completely inside those limits. Standard B values (Figure 131) show that 4 labs had data completely within the control limits, one inside and outside the limits, and 3 labs completely outside these limits. Figures 130 and 131 also show that the results of the ARC-CPA method were frequently located within the warning limits, while the results of other methods were all outside the control limits.

The statistical data for Ni in sludges A to F are shown in Table 14, and their control charts in Figures 132 to 137. Table 14 shows that results (above detection) were reported for all sludges, with number of analyses varying from 25 to 39 samples. The mean Ni values ranged from 9.9 mg/kg for sludge A to 51 for sludge B. The coefficient of variation of the sludges varied from 21 to 72% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 132 to 137 indicate that all the reported Ni data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Ni data shows good recovery of most standards and some agreement between the different labs and for the different methodologies used (except for Standards A and B). The variability seems to be dependent on the number of analyses performed and the Ni concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.1.14 Pb

Eight labs reported Pb in the ICP standard (Figure 138), all but three labs were within the control limits. The three outlier labs were lower than the low control limit. Nine labs reported Pb for the digestate 3 samples (Figure 139) and eight labs for the digestate 4 samples

Figure 128. Mean and Confidence Intervals for Ni in Digestate 3

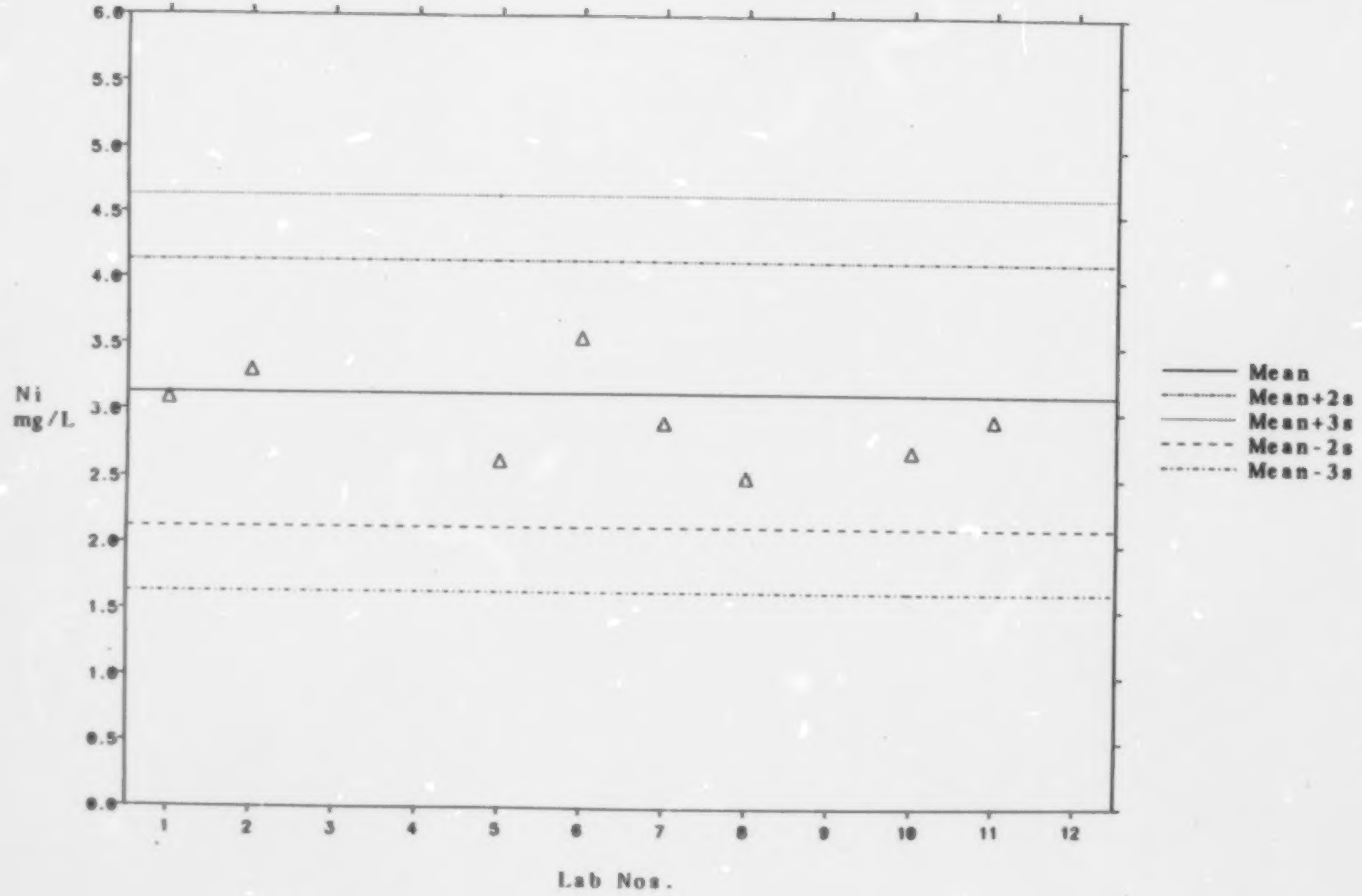


Figure .29. Mean and Confidence Intervals for Ni in Digestate 4

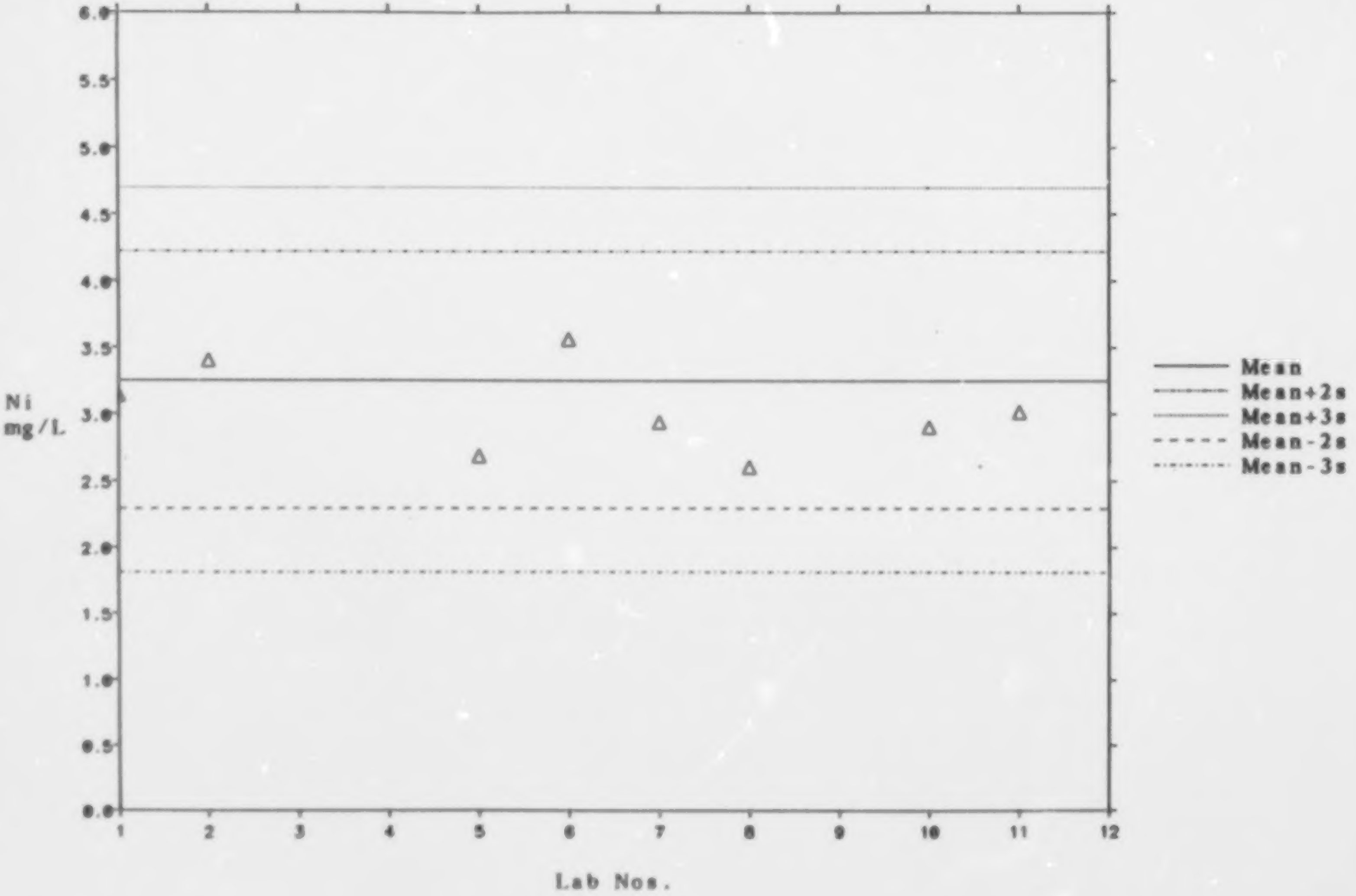


Figure 130. Mean and Confidence Intervals for Ni in Standard A

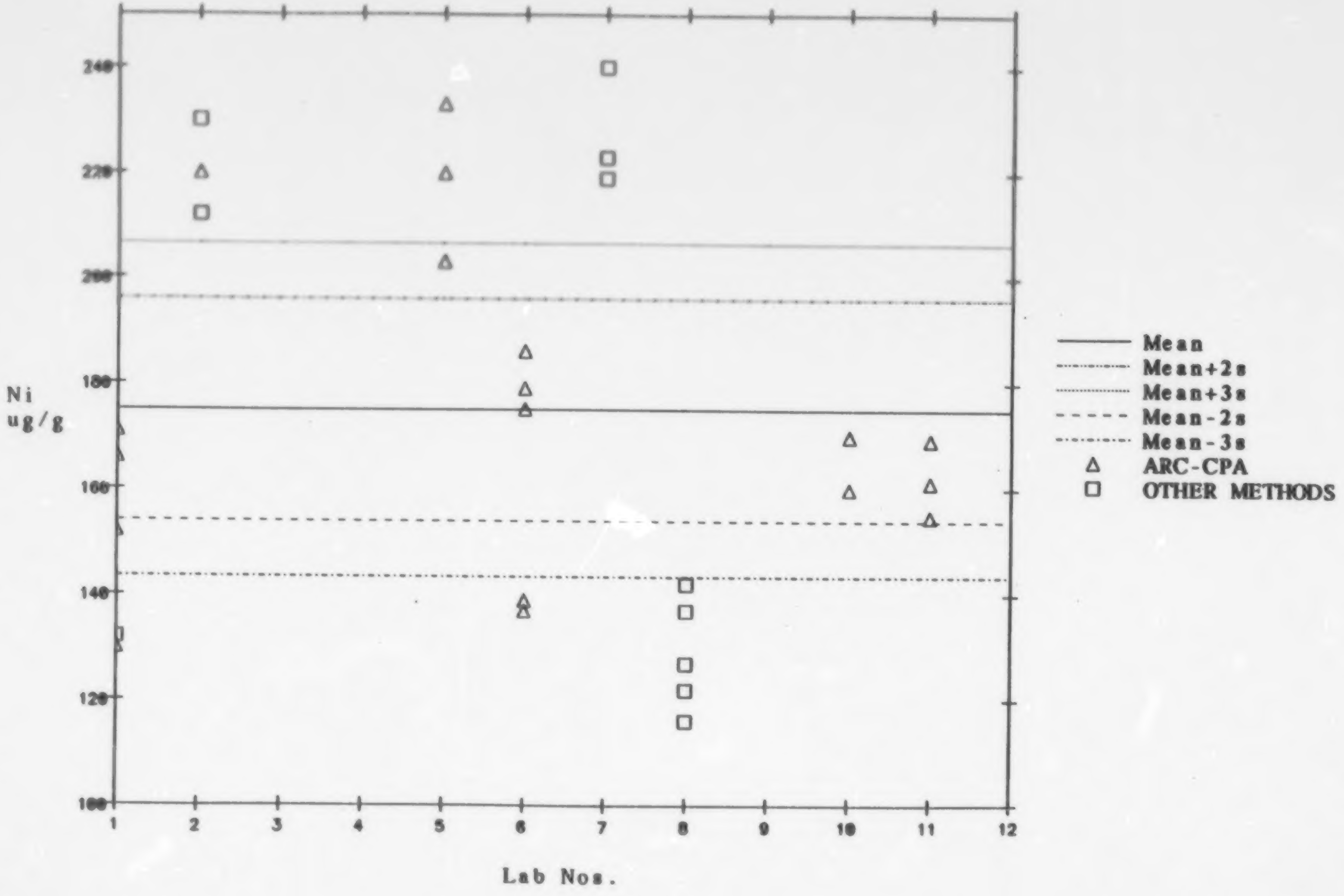


Figure 131. Mean and Confidence Intervals for Ni in Standard B

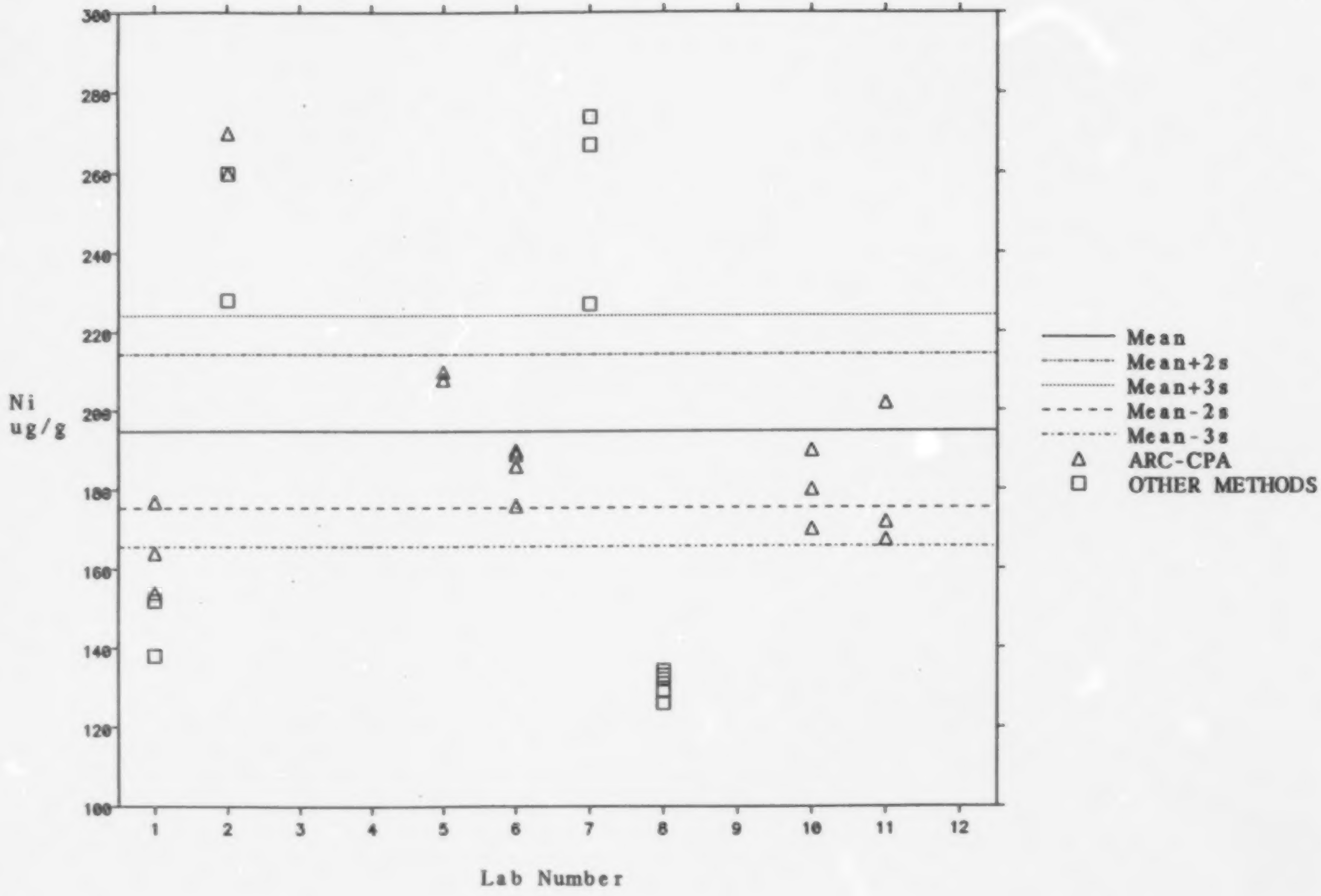


Table 14. Statistical Parameters for Ni (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	25.00	38.00	26.00	39.00	26.00	29.00
Sum	248.31	1939.40	514.37	3171.71	737.82	789.91
Mean	9.93	51.04	19.78	81.33	28.38	27.24
SEM	1.43	1.71	1.91	3.87	1.83	2.36
Median	9.00	48.00	15.00	71.15	26.00	21.00
Variance	50.77	111.25	94.92	583.33	87.41	161.47
StDev	7.13	10.55	9.74	24.15	9.35	12.71
Maximum	37.20	83.10	35.60	133.00	58.52	59.00
Minimum	2.18	33.00	8.33	42.30	14.00	14.00
Range	35.02	50.10	27.27	90.70	44.52	45.00
Skewness	2.56	0.87	0.55	0.65	1.56	1.38
Kurtosis	8.71	1.17	-1.57	-0.28	3.56	0.78
CV(%)	71.80	20.67	49.24	29.69	32.94	46.66

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 132. Analysis of Ni in Waste A

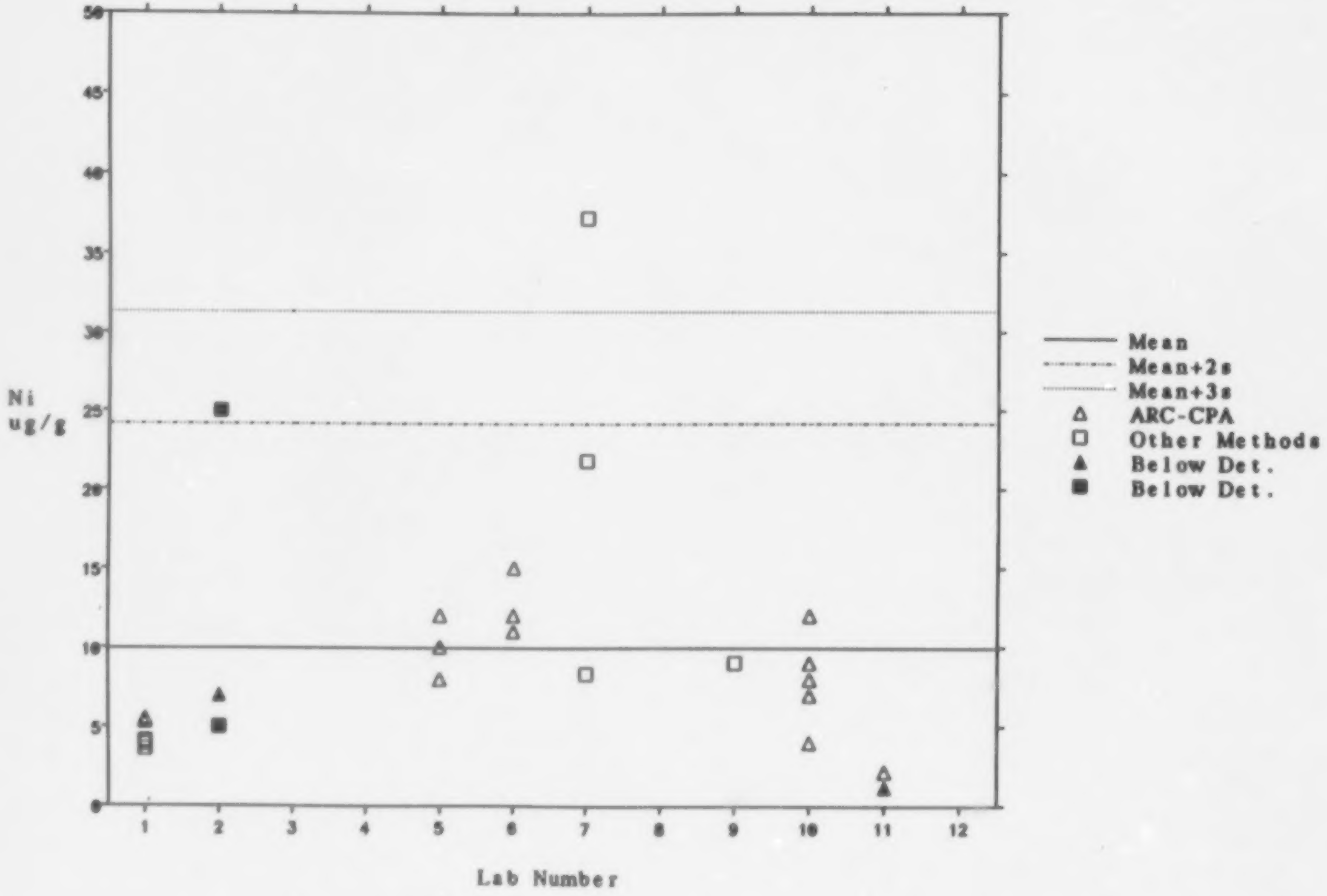


Figure 133. Analysis of Ni in Sludge B Samples

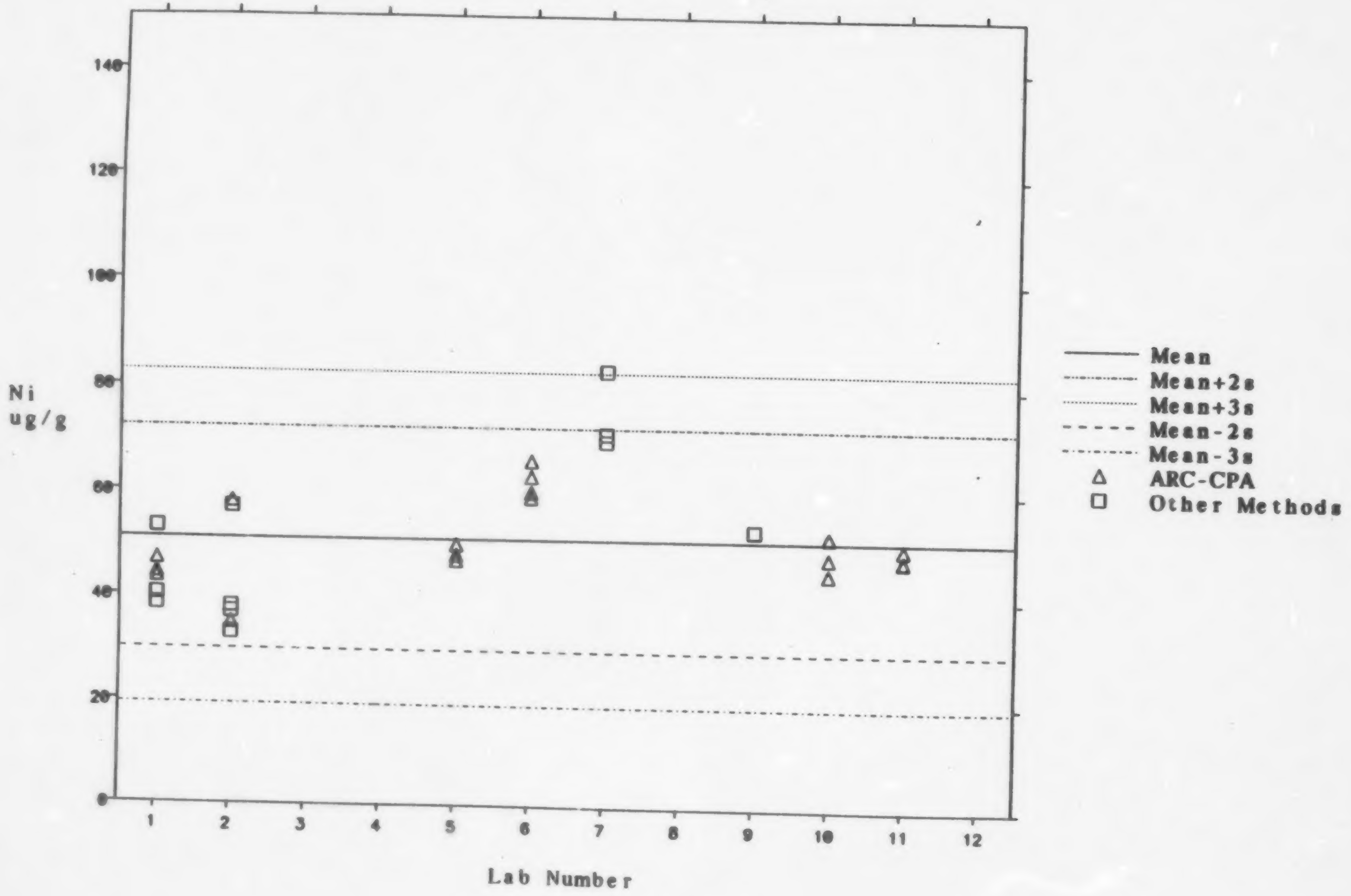


Figure 134. Analysis of Ni in Sludge C

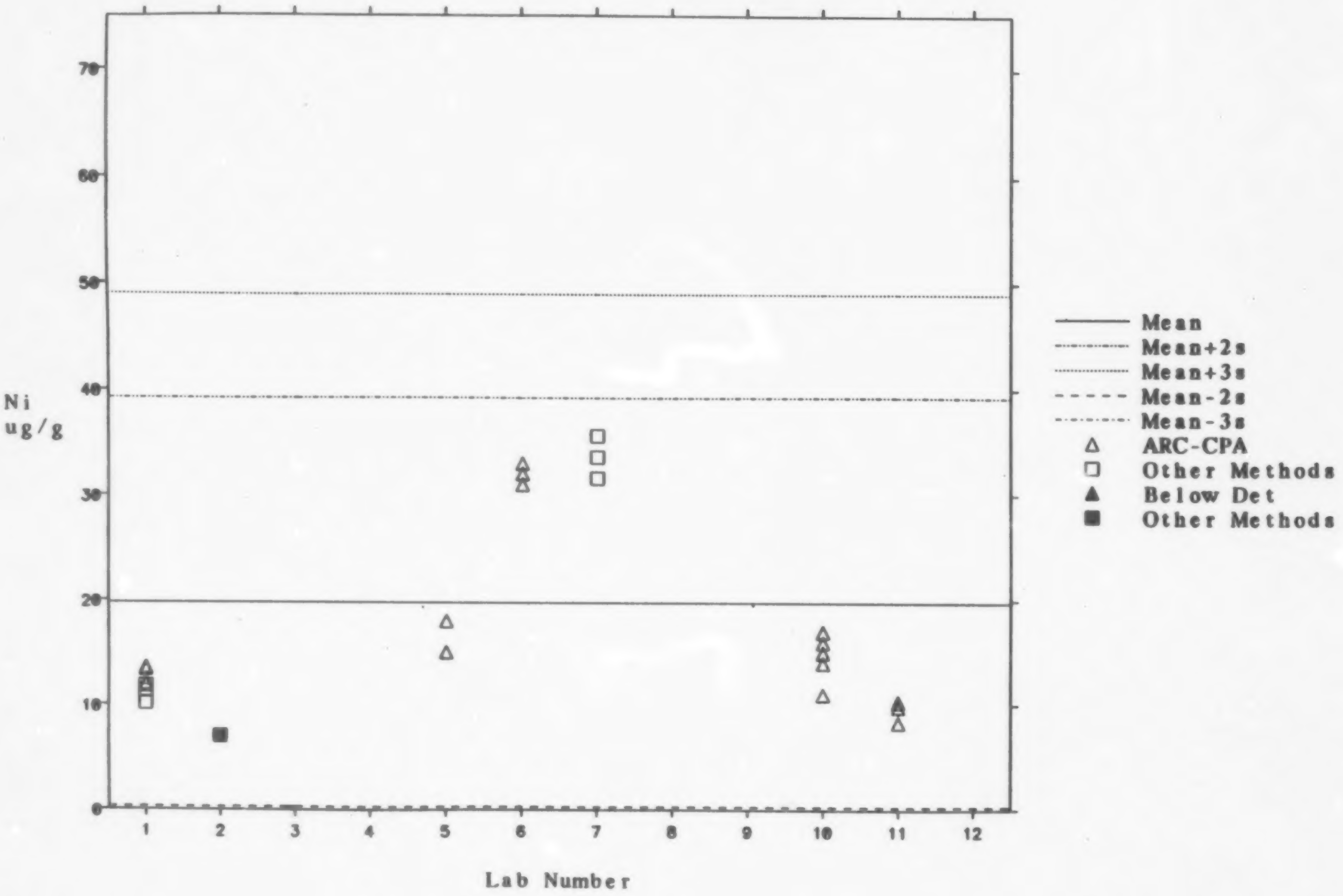


Figure 135. Analysis of Ni in Sludge D

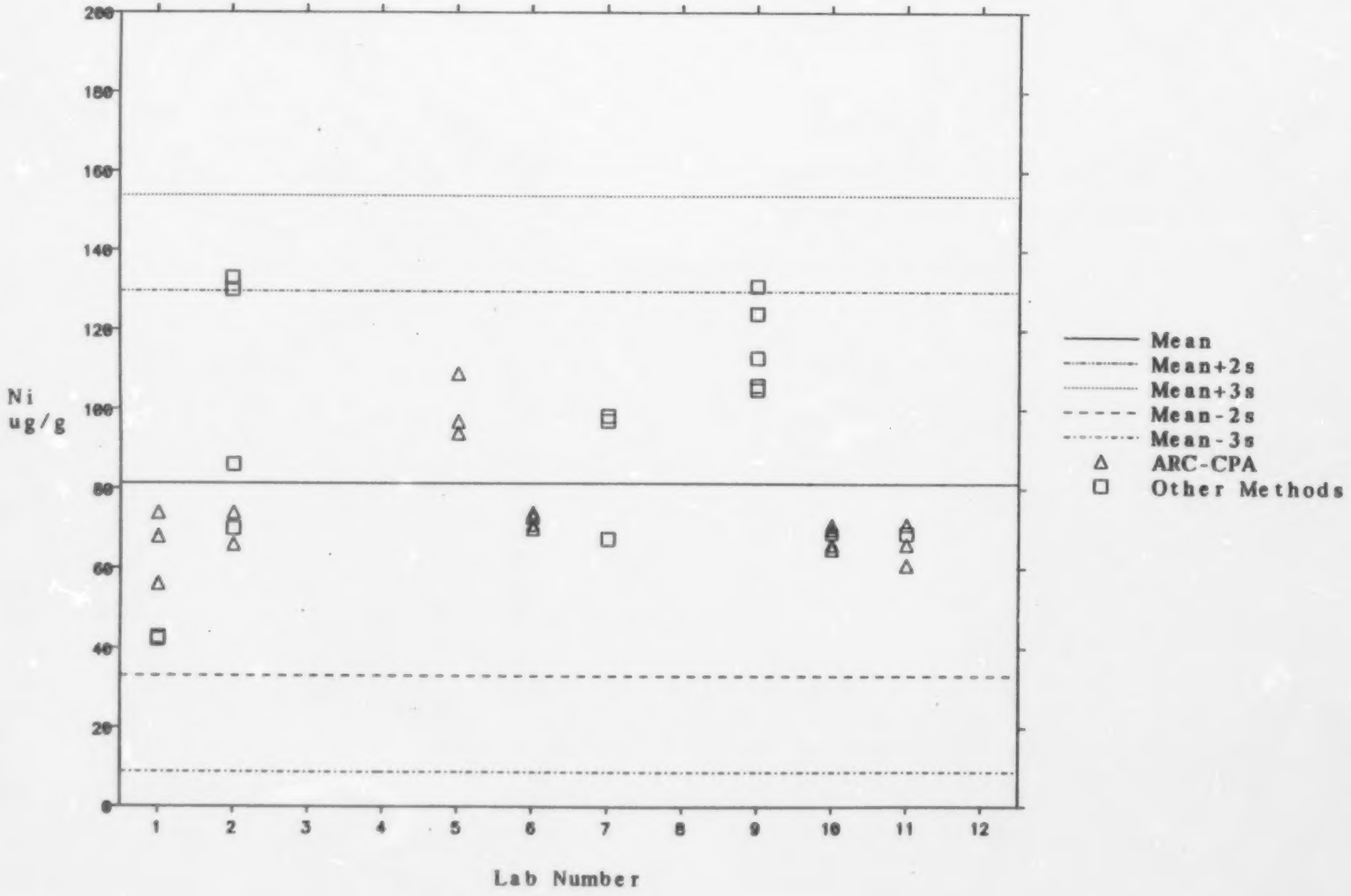


Figure 136. Analysis of Ni in Sludge E

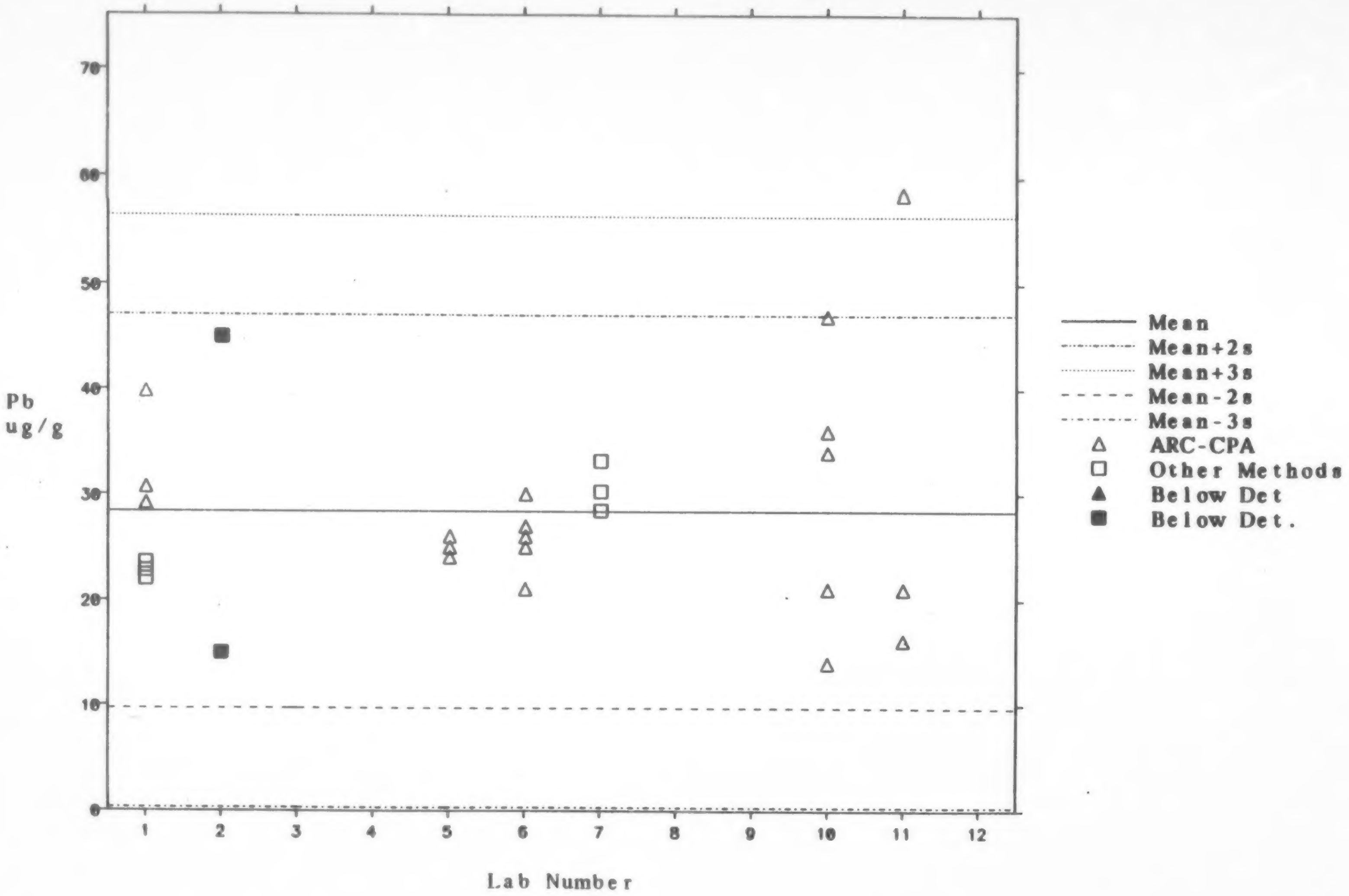


Figure 137. Analysis of Ni in Sludge F

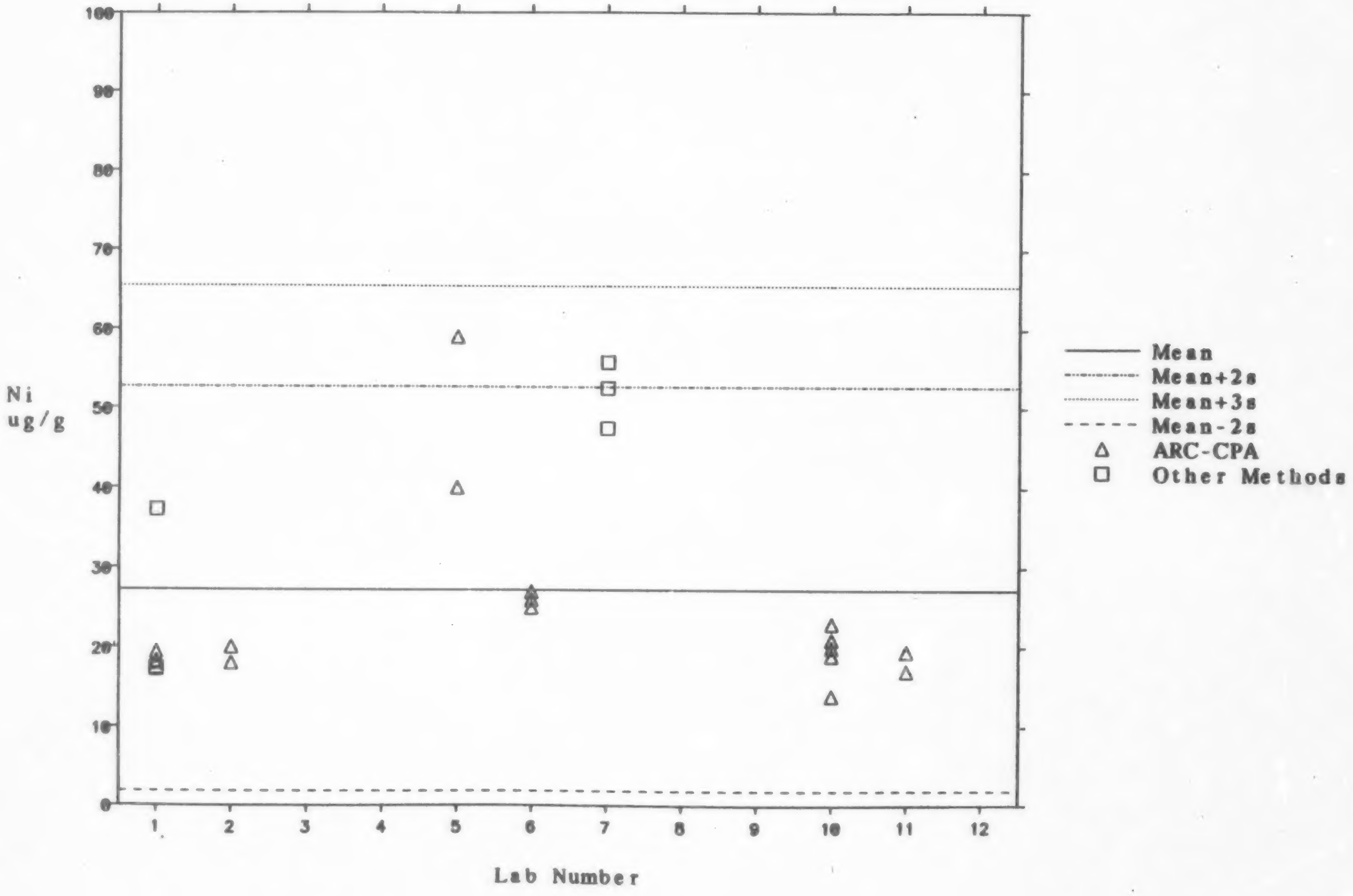


Figure 138. Mean and Confidence Intervals for Pb in an ICP Standard

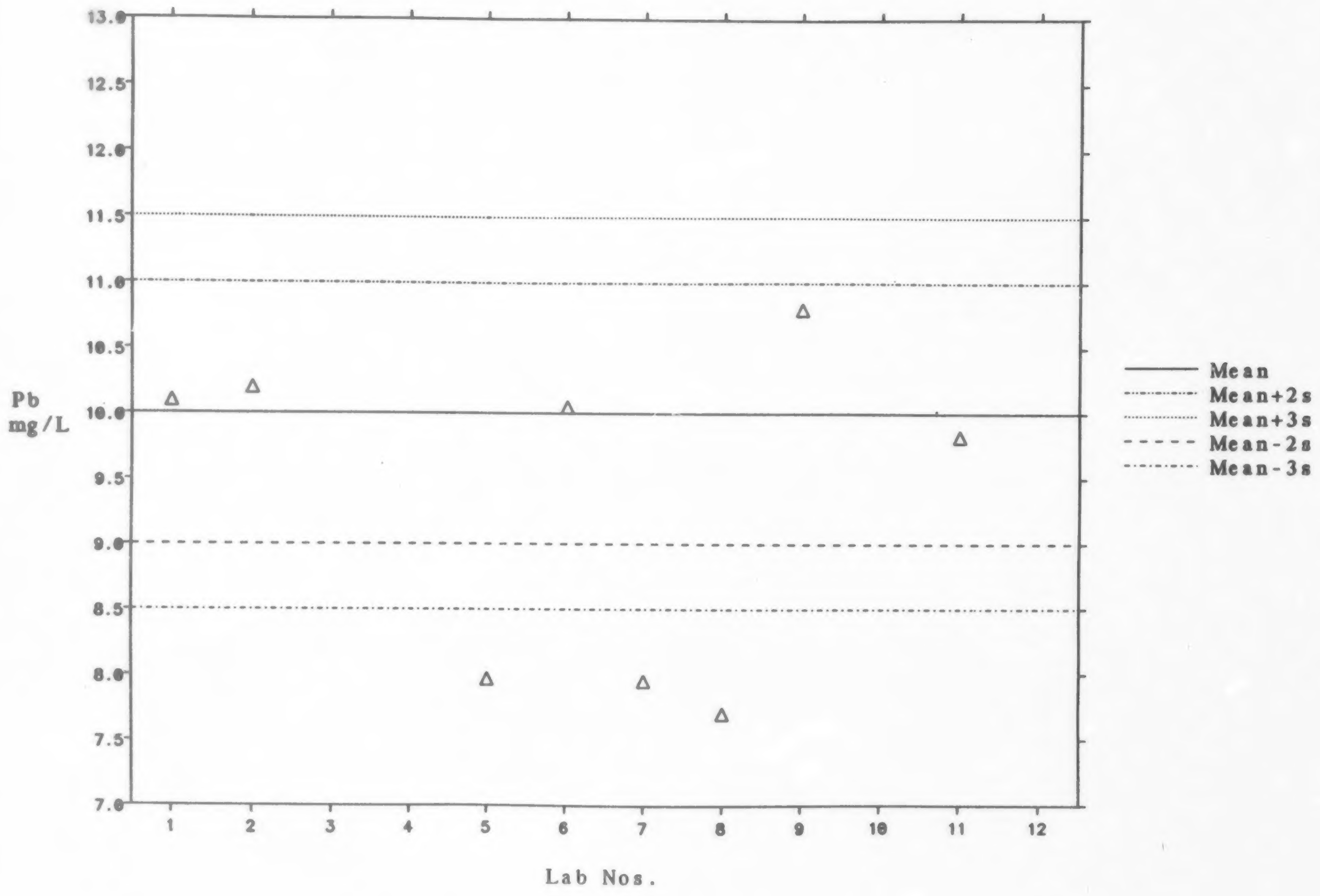
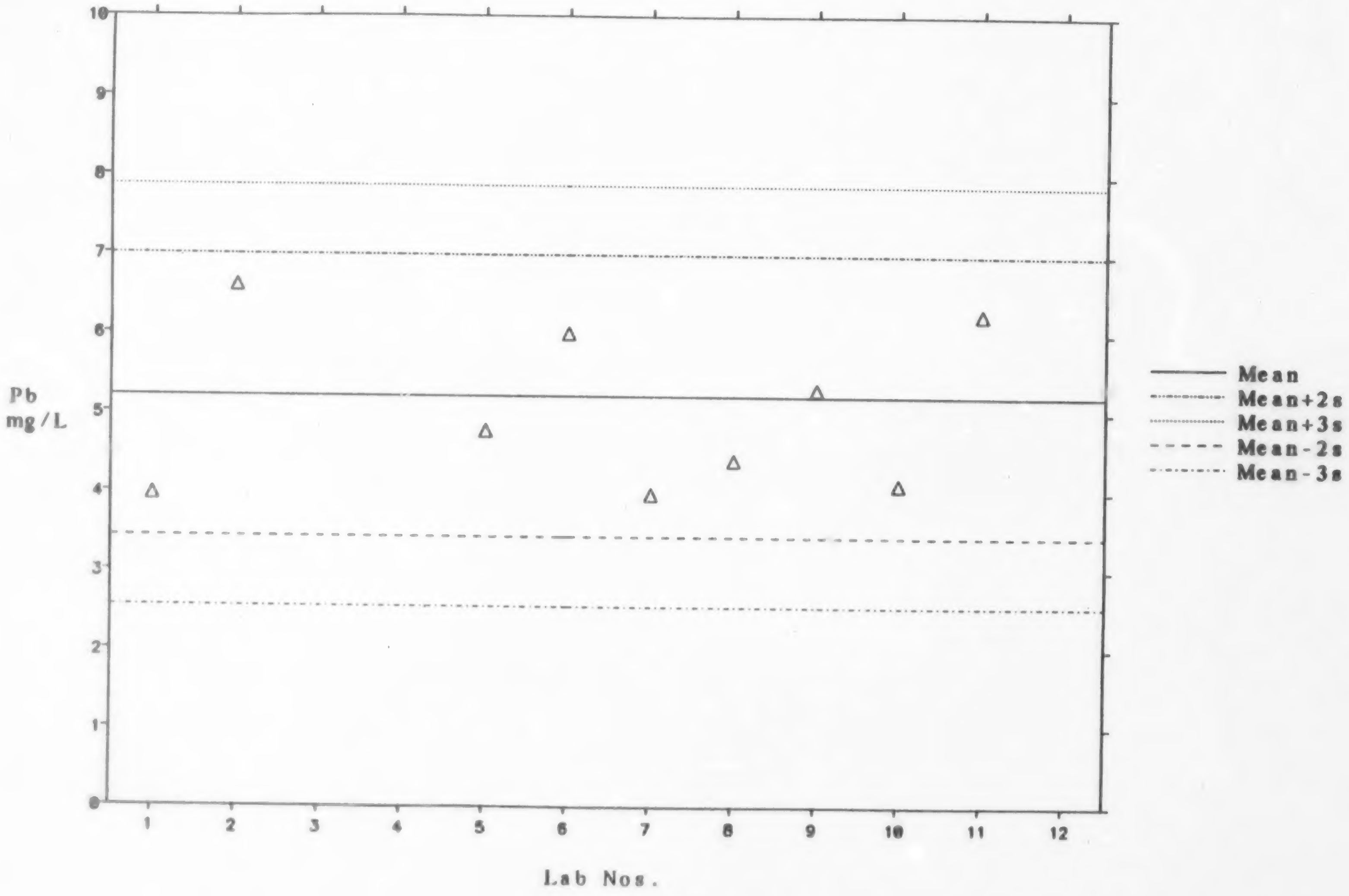


Figure 139. Mean and Confidence Intervals for Pb in Digestate 3



(Figure 140). Digestate 3 data (Figure 139) were all within the warning limits, and all but two labs were inside the warning limits of digestate 4 (Figure 140). The digestate data shows very good recovery and agreement between the participating labs. Eight labs reported Pb for the standard A and B samples (Figures 141 and 142). Figure 141 shows that all labs (except one) reported data within Standards A's control limits. Standard B values (Figure 142) show that none of the labs had data completely within the warning limits, with 4 labs reporting data completely outside the control limits. Figures 141 and 142 also show that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Pb in sludges A to F are shown in Table 15, and their control charts in Figures 143 to 148. Table 15 shows that results (above detection) were reported for all sludges, with number of analyses varying from 6 to 44 samples. The mean Pb values ranged from 9.8 mg/kg for sludge E to 1210 for sludge B. The coefficient of variation of the sludges varied from 12 to 86% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 143 to 148 indicate that most of the reported Pb data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Pb data shows good recovery of most standards and some agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed and the Pb concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.1.15 Sb

Only one lab reported Sb in the ICP standard (Figure 149), and this value was significantly higher than the high control limit. Three labs reported Sb for the digestate samples (Figures 150 and 151). Digestate 3 data (Figure 150) were all outside the control limits, while digestate 4 (Figure 151) values were all inside the warning limits. Five labs reported Sb for the standard A and B samples (Figures 152 and 153). Figure 152 shows that 4 labs reported data within Standards A's control limits and the other one was outside those limits. Standard B values (Figure 153) show that 3 labs had data within the control limits, with the other 2 labs reporting data outside these limits. Figures 152 and 153 show that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Sb in sludges A to F are shown in Table 16, and their control charts in Figures 154 to 159. Table 16 shows that results (above detection) were reported for all sludges (except E), with number of analyses varying from 5 to 7 samples. The mean Sb values ranged from 0.89 mg/kg for sludge A to 84.3 for sludge C. The coefficient of variation of the sludges varied from 41 to 151% and it seems to be inversely related to the number of reported results.

Figure 140. Mean and Confidence Intervals for Pb in Digestate 4

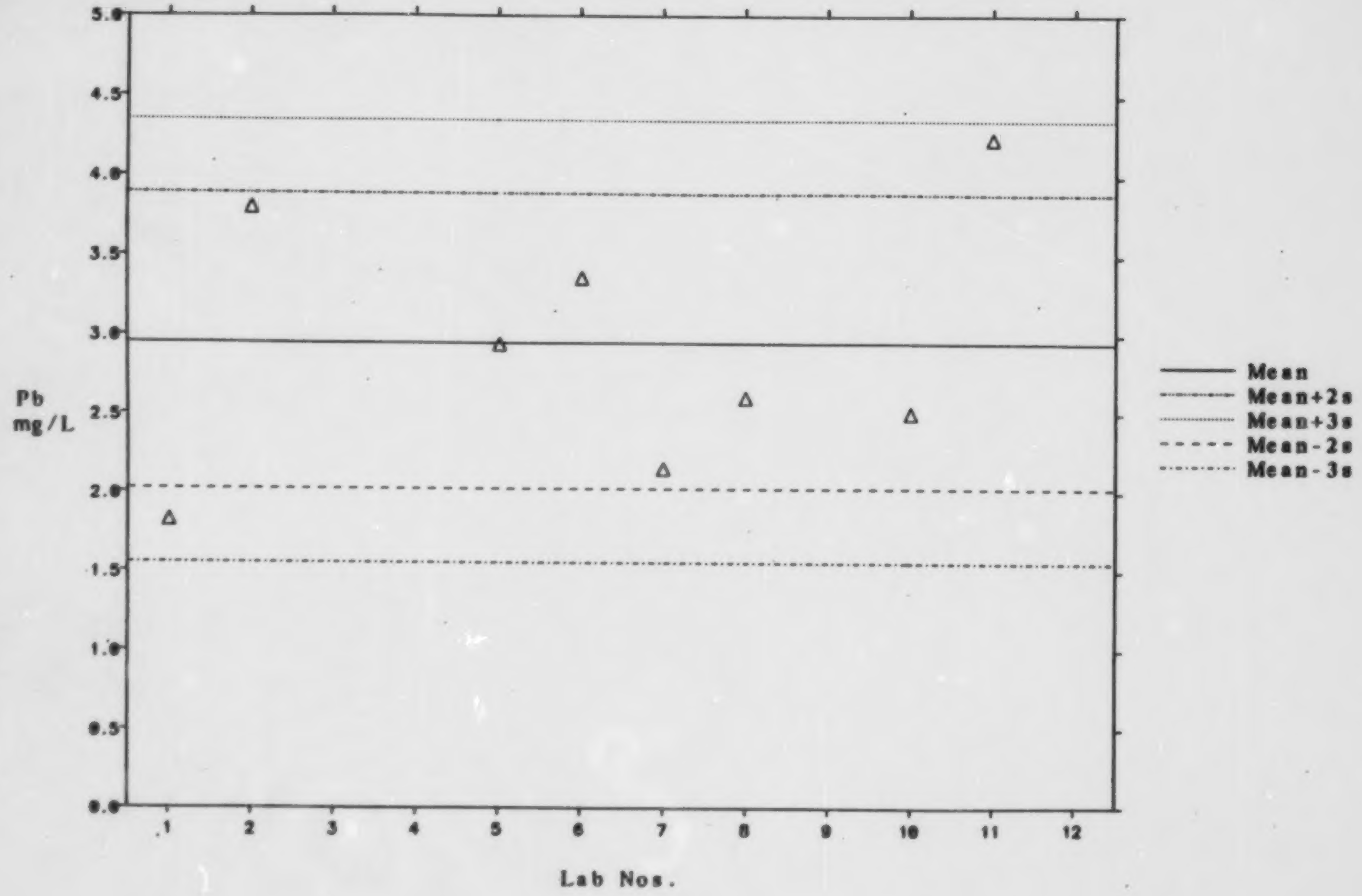


Figure 141. Mean and Confidence Intervals for Pb in Standard A

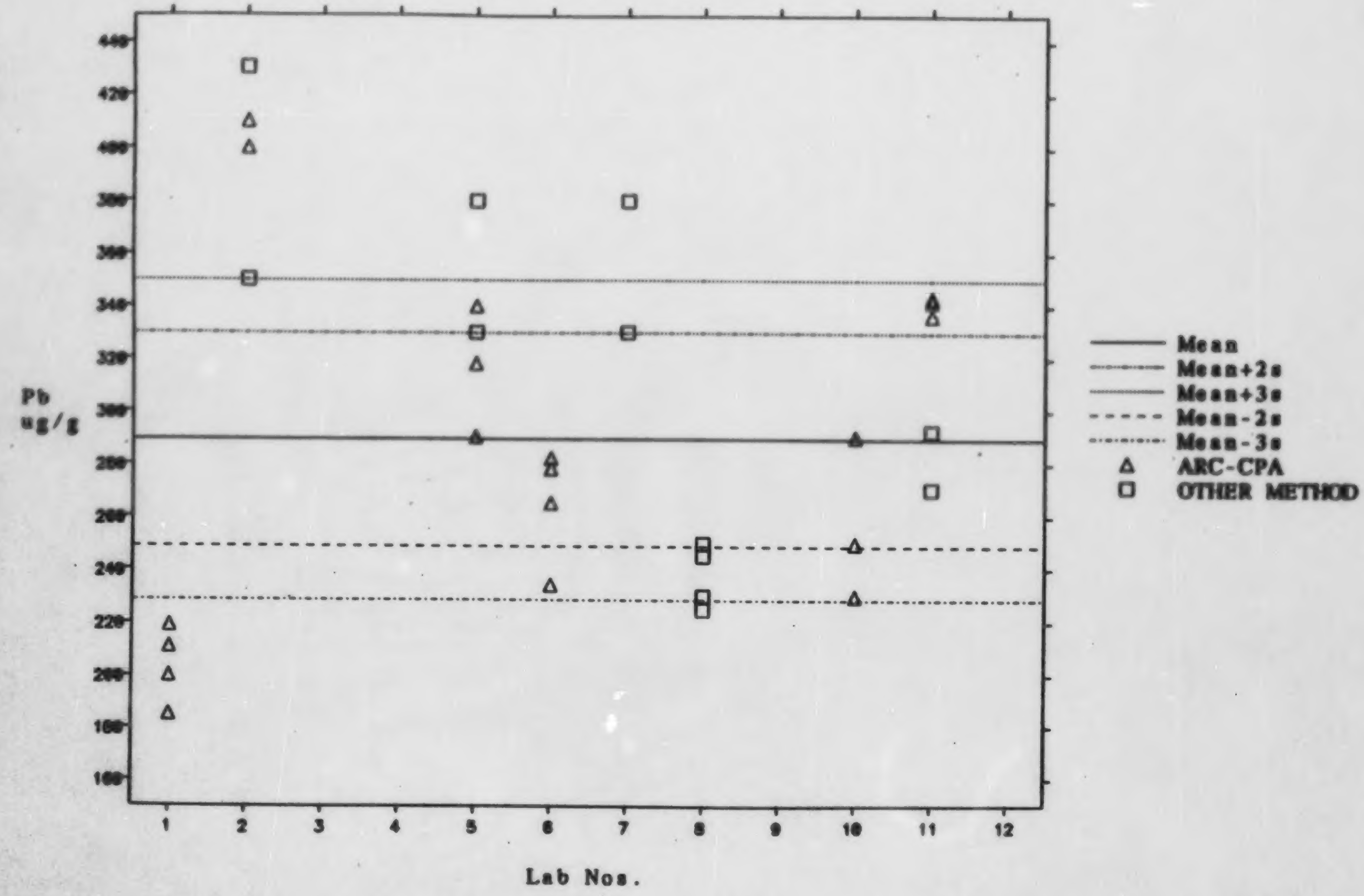


Figure 142. Mean and Confidence Intervals for Pb in Standard B

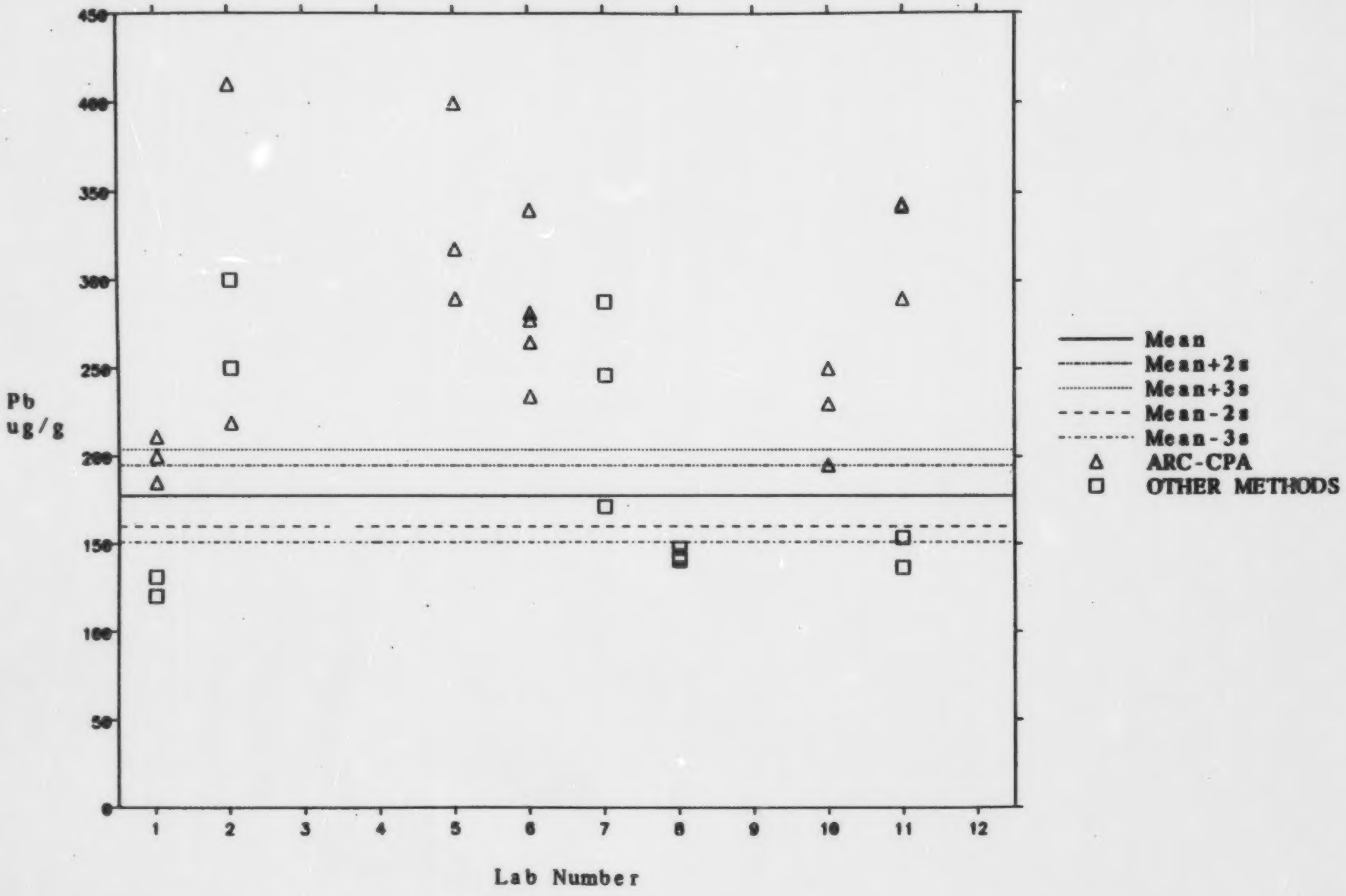
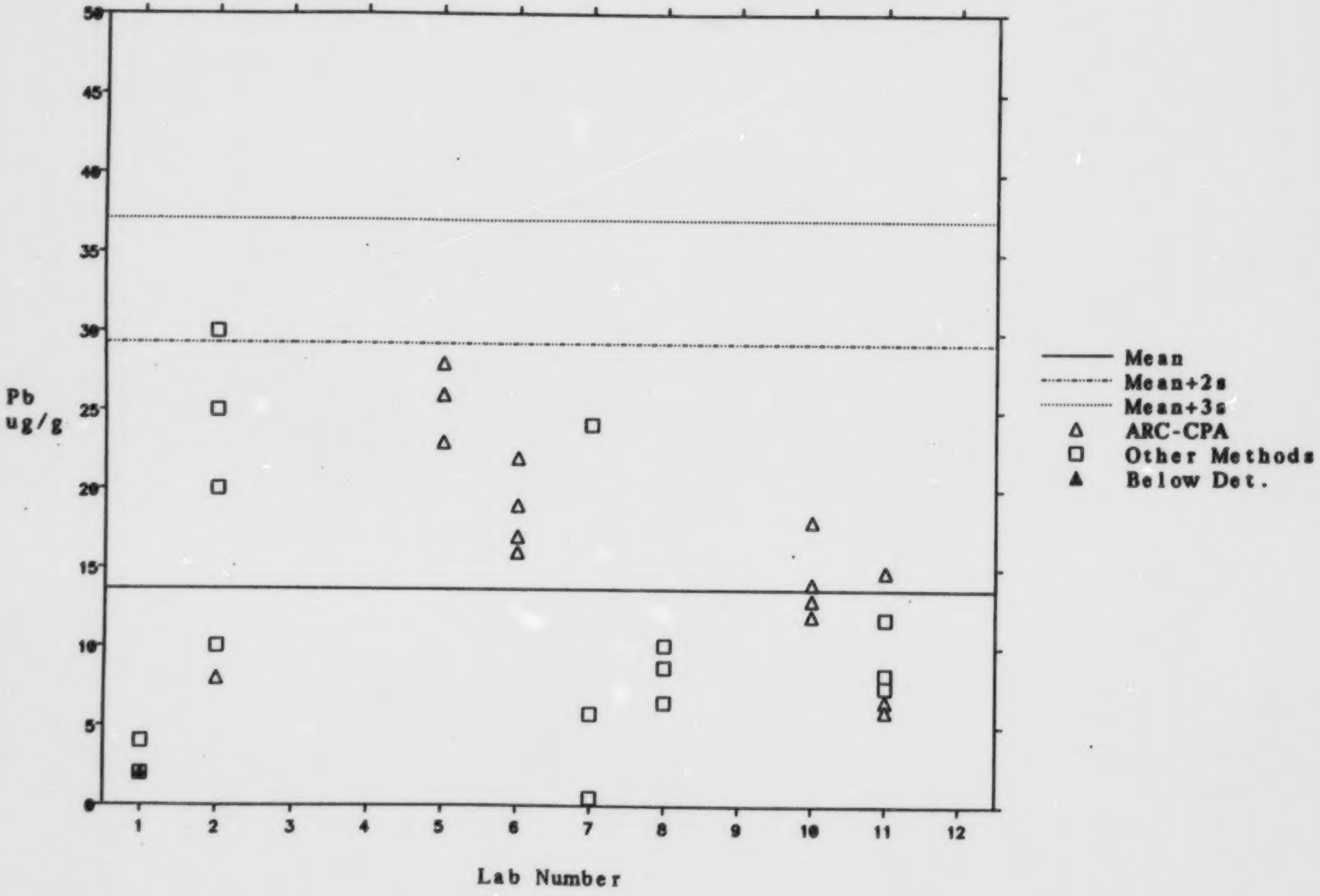


Table 15. Statistical Parameters for Pb (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	35.00	44	33.00	42.0	6.000	37.0
Sum	478.00	53300	775.00	1340.0	59.000	1830.0
Mean	13.60	1210	23.50	31.9	9.830	49.3
SEM	1.32	22	2.11	3.4	3.470	3.5
Median	12.00	1200	24.00	28.5	6.500	47.0
Variance	61.00	22100	146.00	484.0	72.200	462.0
StDev	7.81	149	12.10	22.0	8.500	21.5
Maximum	30.00	1570	44.00	105.0	24.000	90.0
Minimum	0.51	1000	2.00	7.0	2.000	2.0
Range	29.50	570	42.00	98.0	22.000	88.0
Skewness	0.37	6e-01	0.02	2.1	1.120	-0.3
Kurtosis	-0.78	-5e-01	-0.89	4.8	0.095	-0.2
CV(%)	57.20	12	51.50	68.9	86.400	43.6

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 143. Analysis of Pb in Waste A



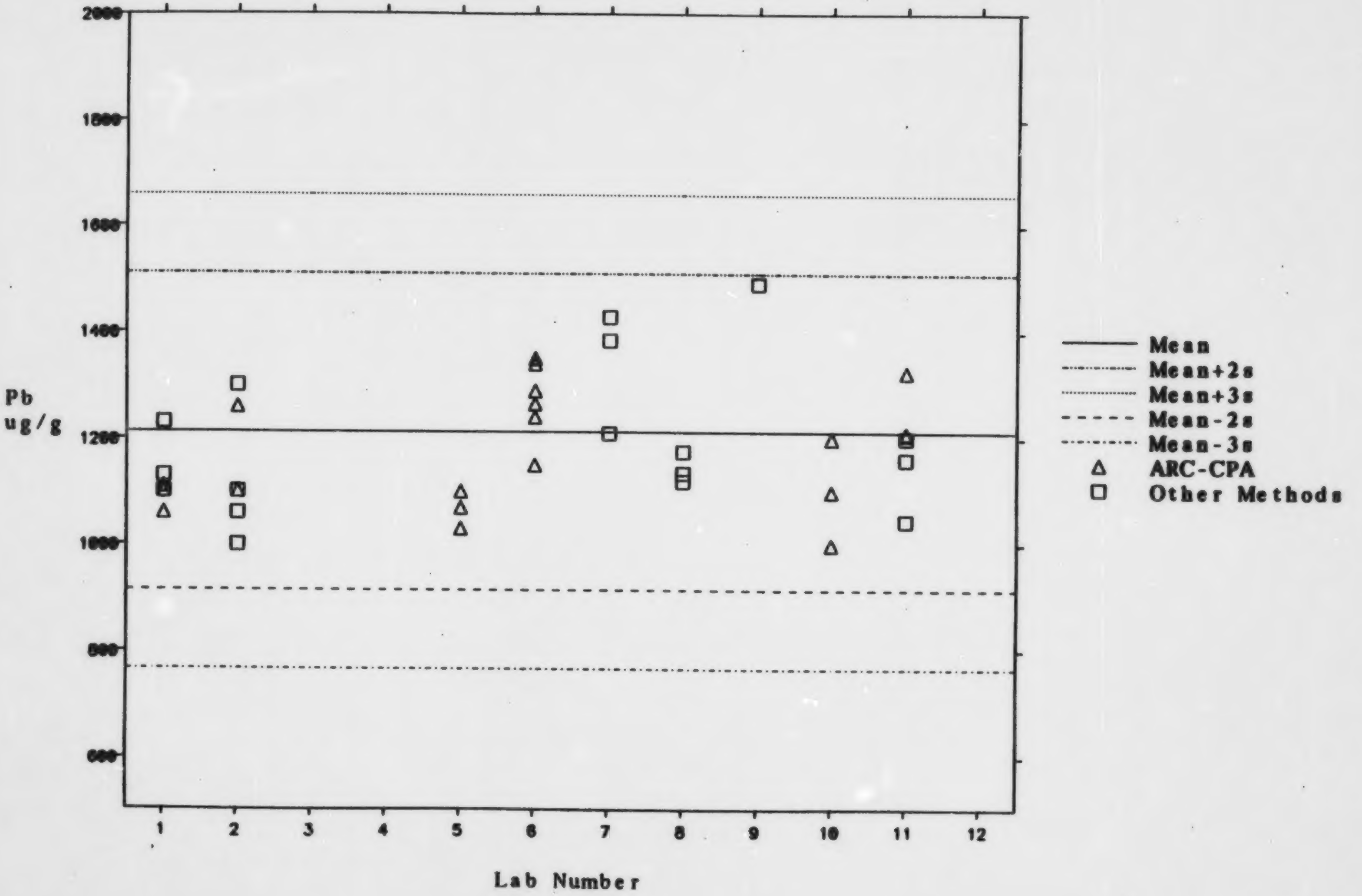


Figure 144. Analysis of Pb in Sludge B Samples

Figure 145. Analysis of Pb in Sludge C

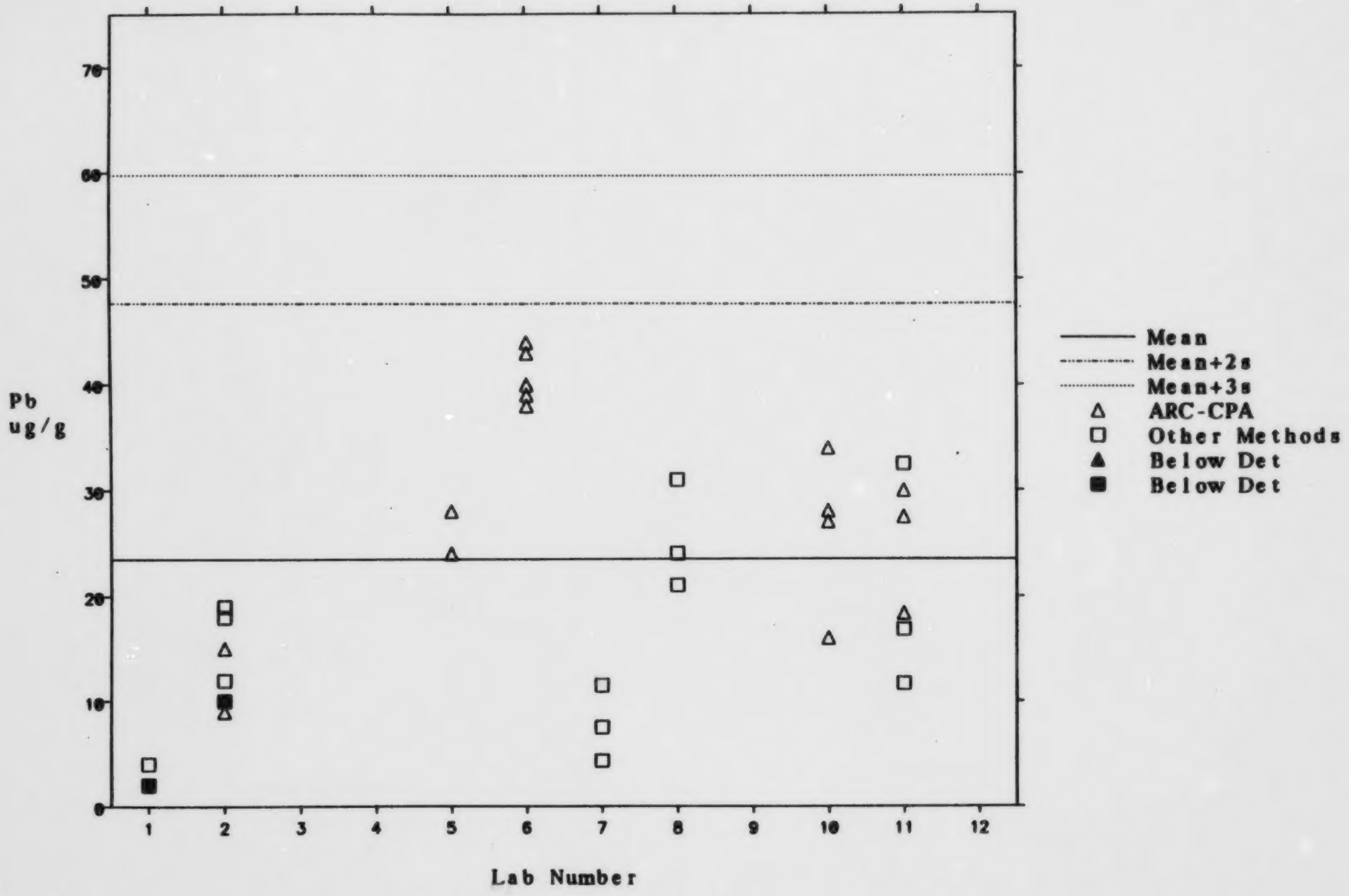


Figure 146. Analysis of Pb in Sludge D

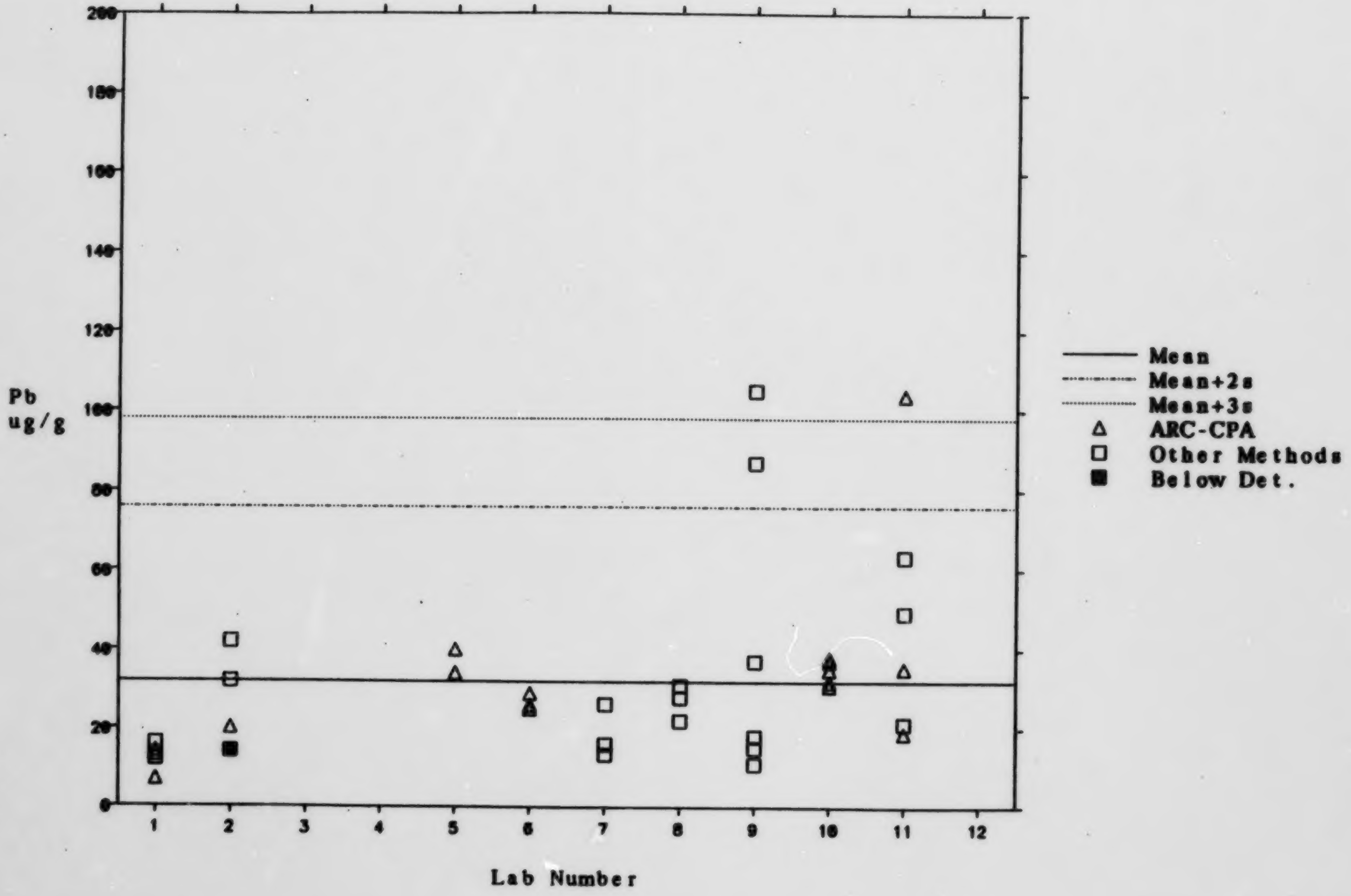


Figure 147. Analysis of Pb in Sludge E

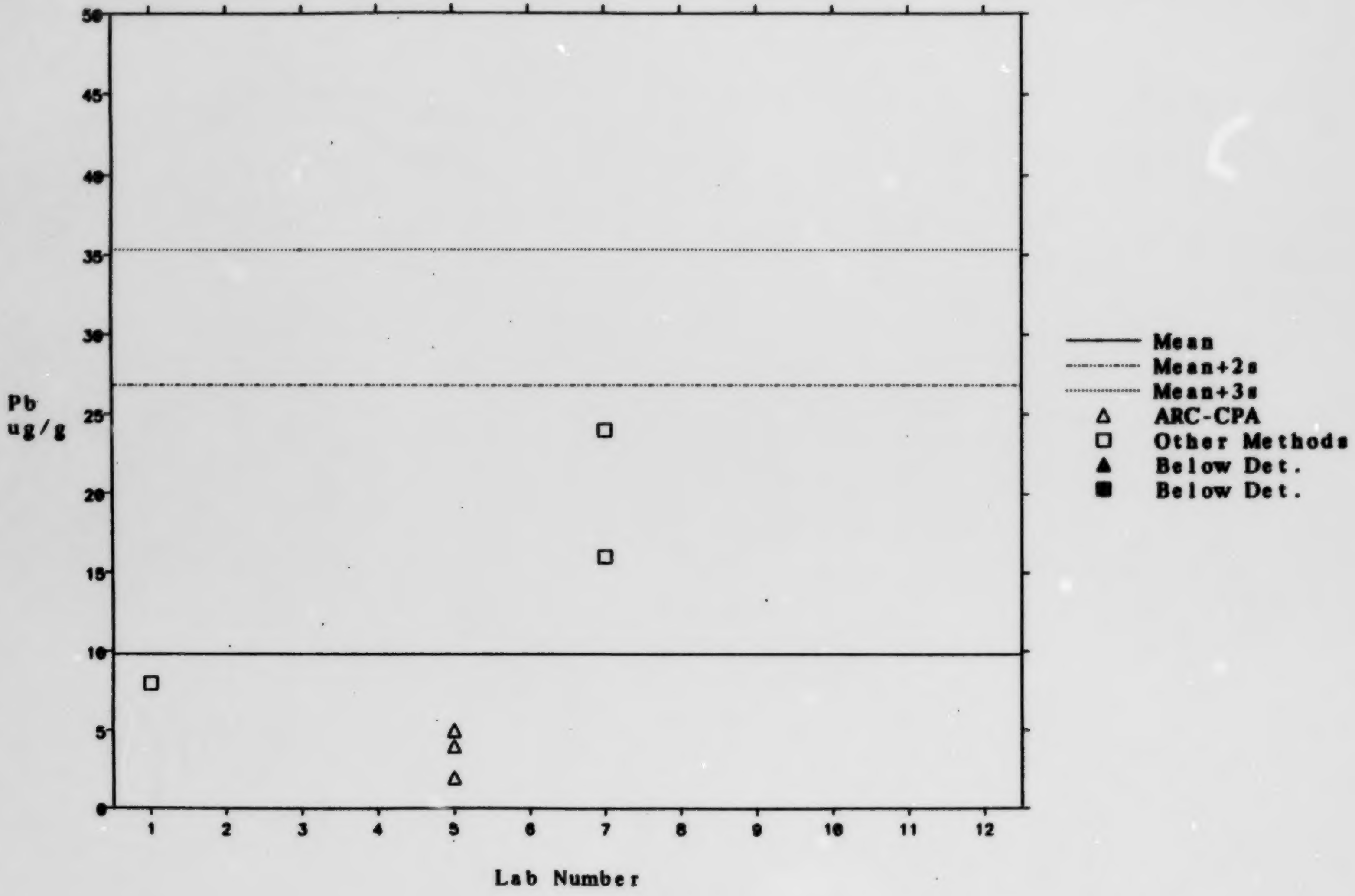


Figure 148. Analysis of Pb in Sludge F

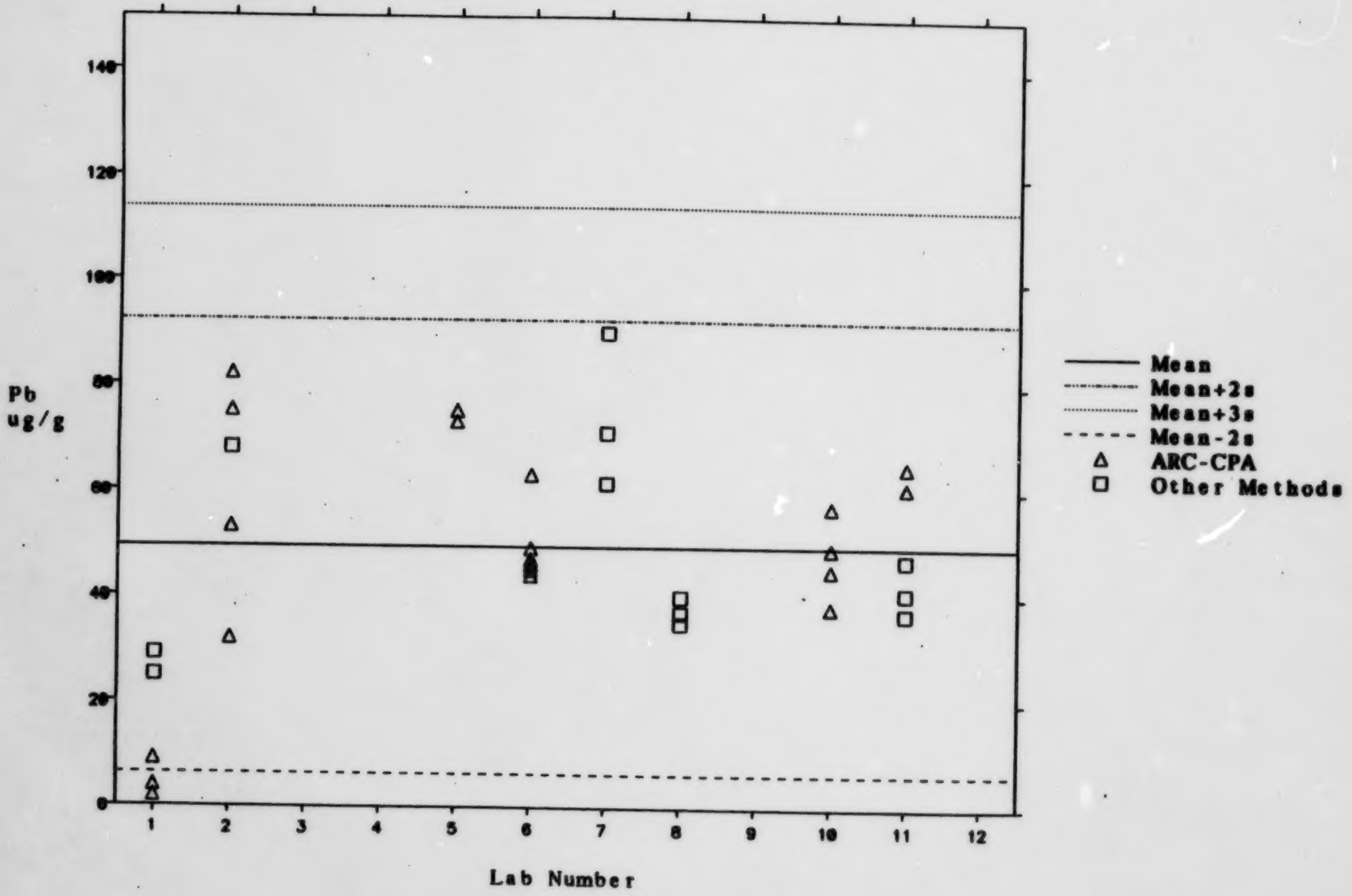


Figure 149. Mean and Confidence Intervals for Sb in an ICP Standard

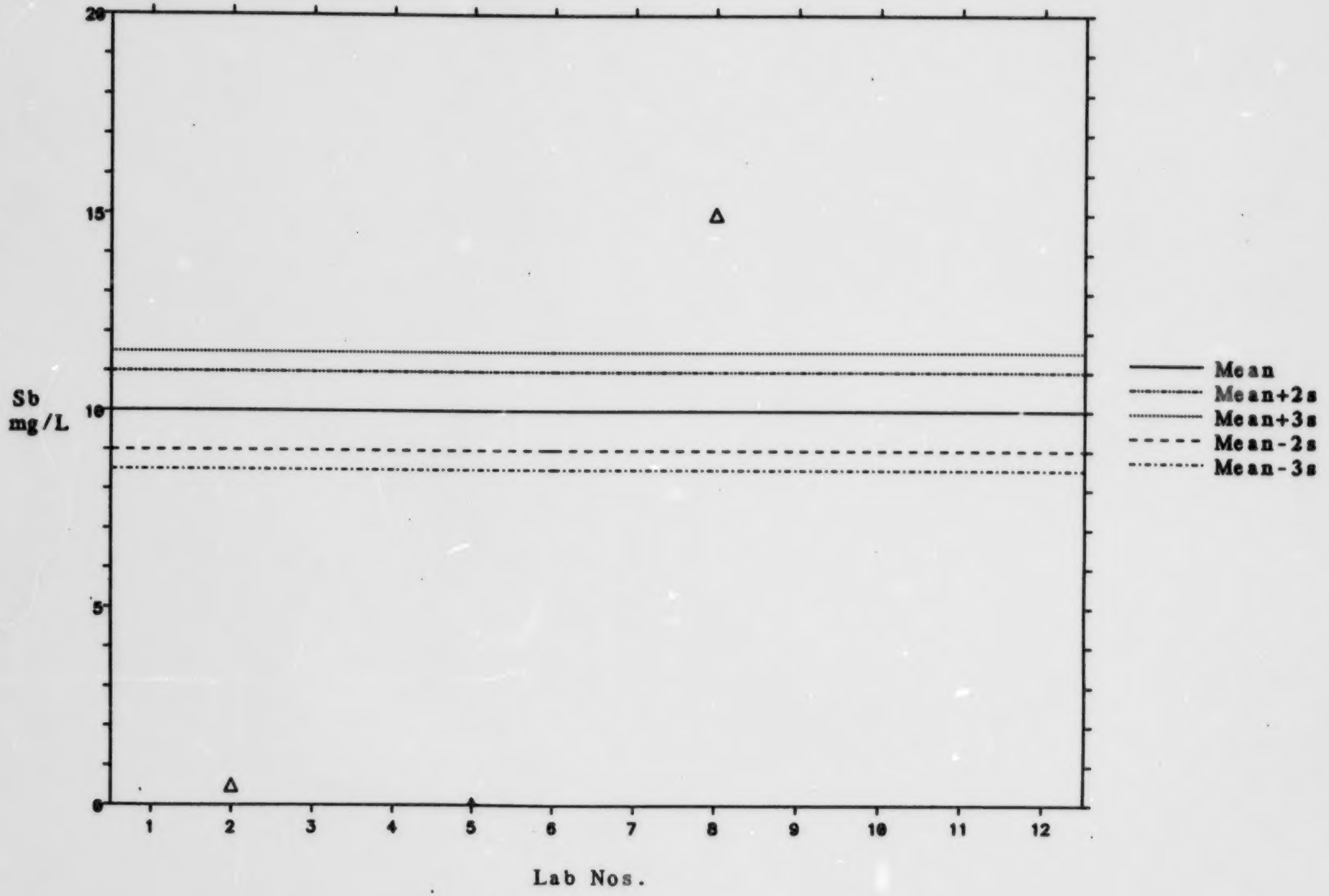
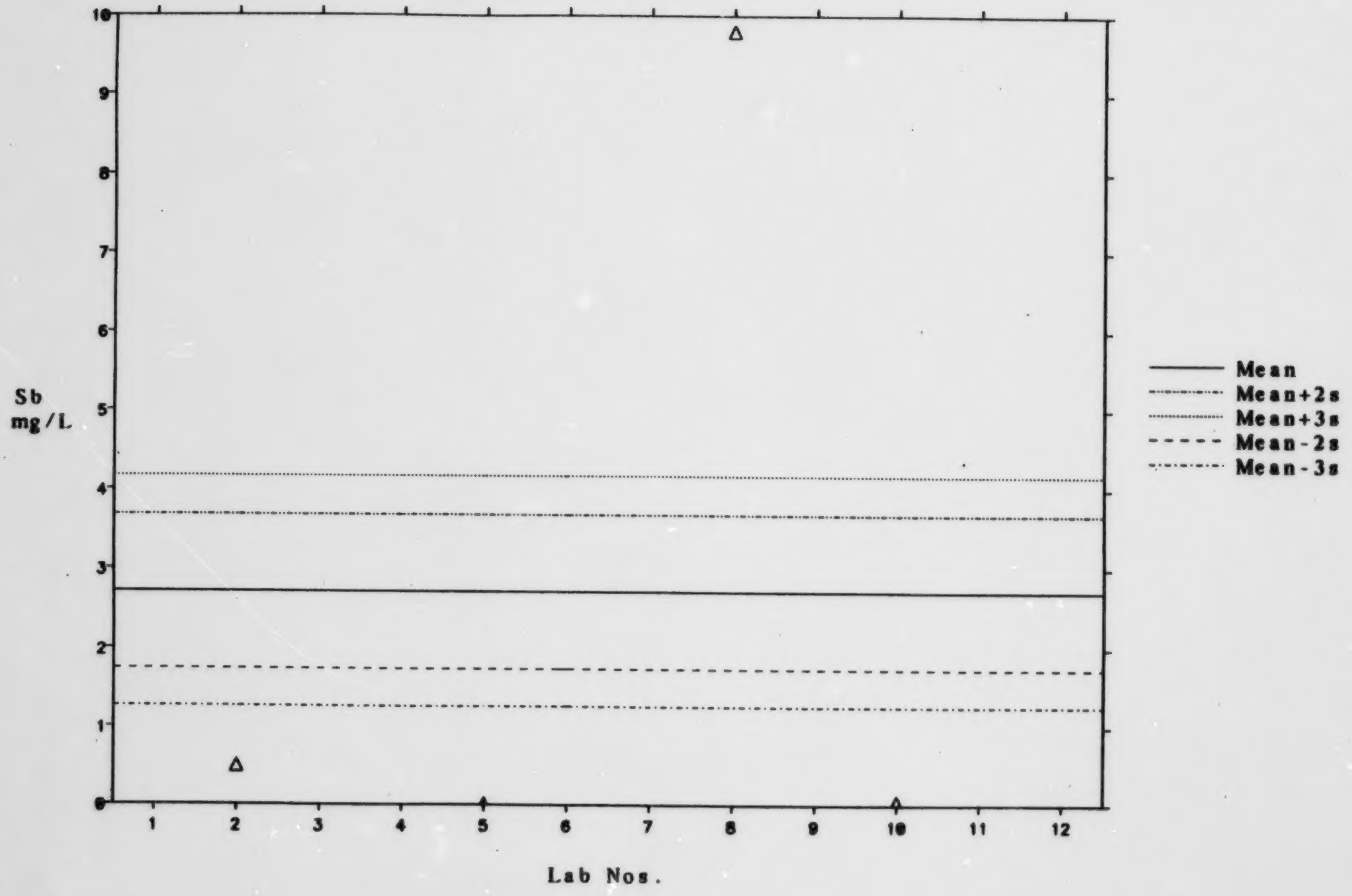


Figure 150. Mean and Confidence Intervals for Sb in Digestate 3



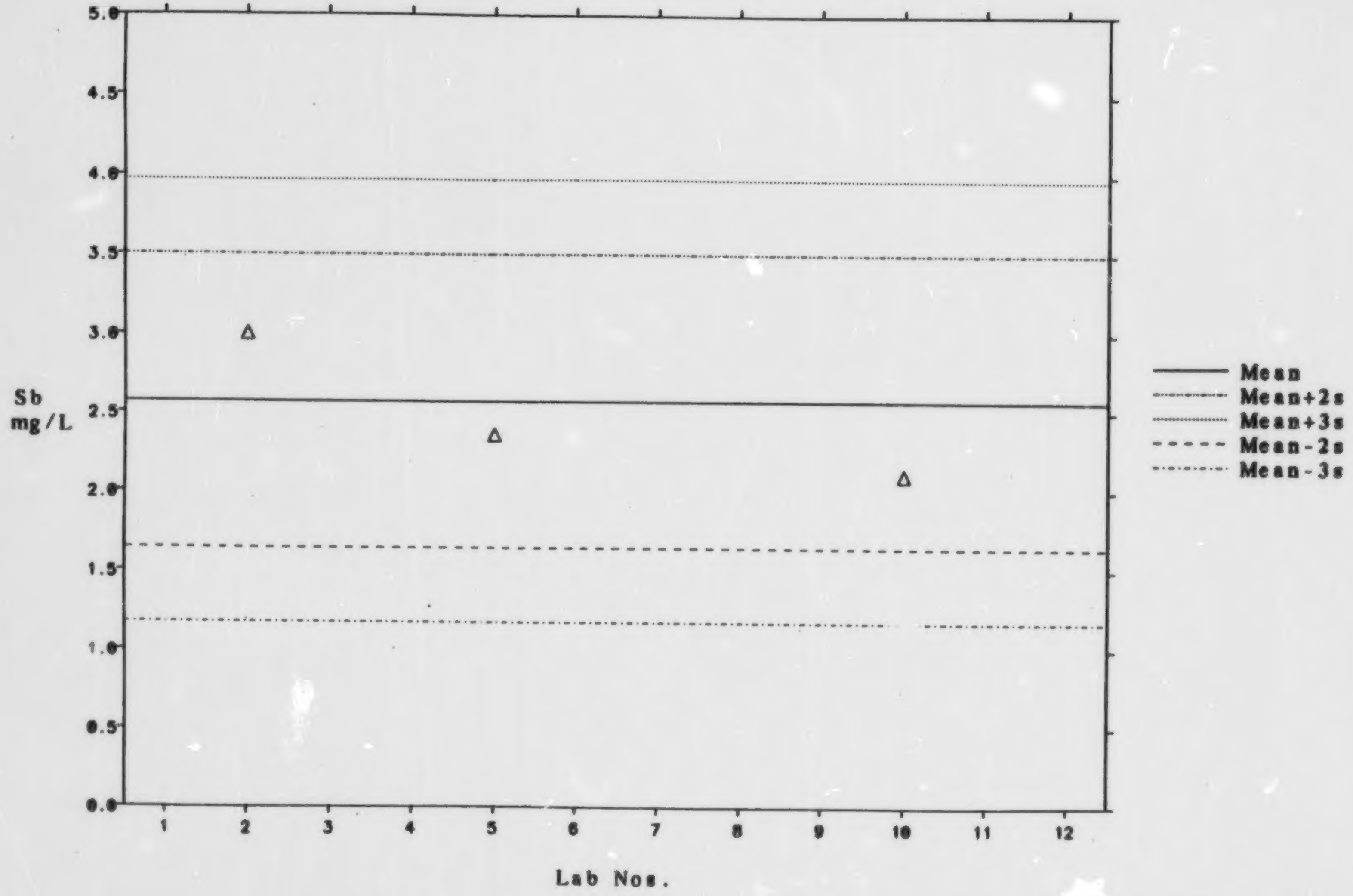


Figure 151. Mean and Confidence Intervals for Sb in Digestate 4

Figure 152. Mean and Confidence Intervals for Sb in Standard A

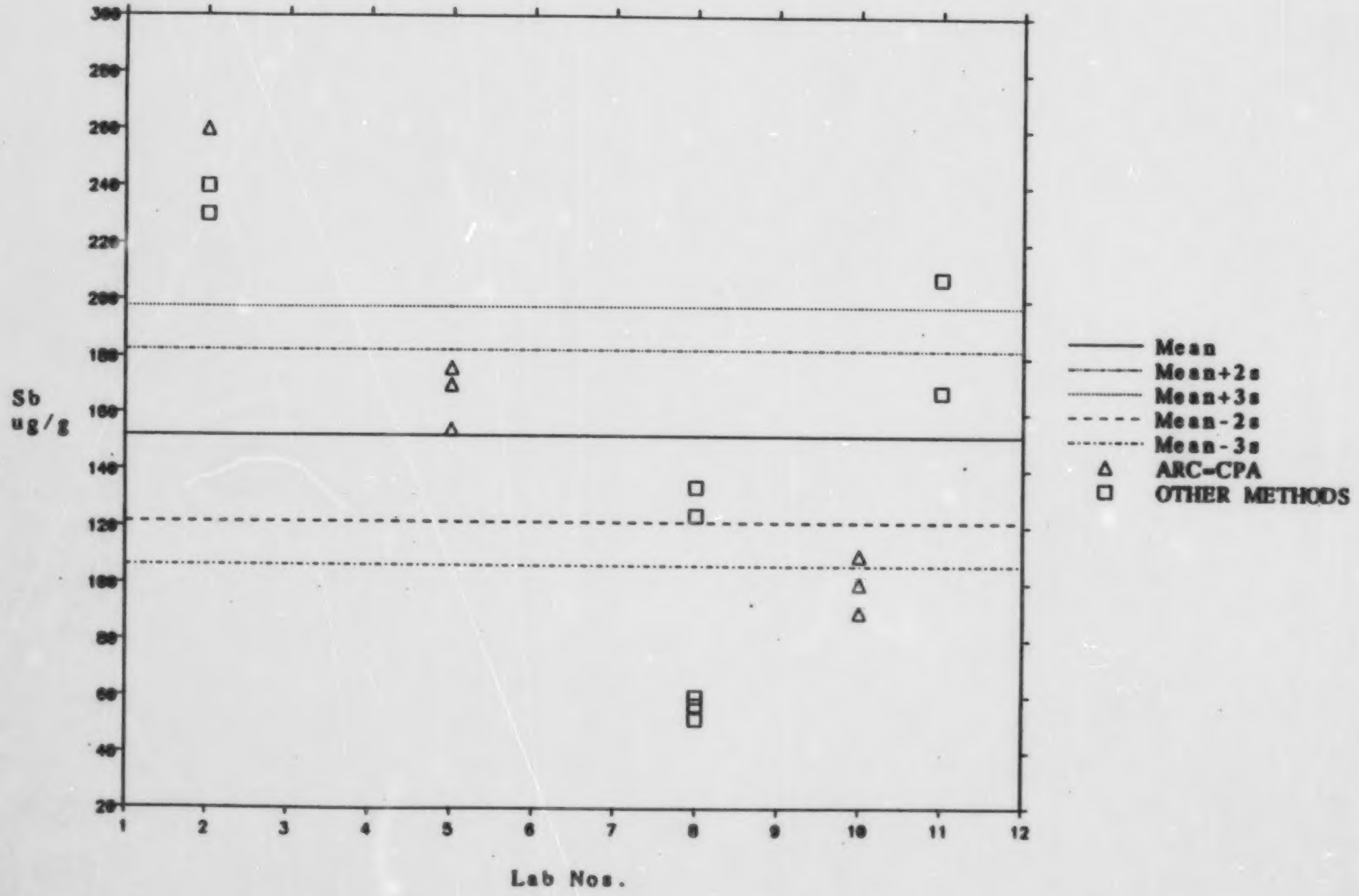


Figure 153. Mean and Confidence Intervals for Sb in Standard B

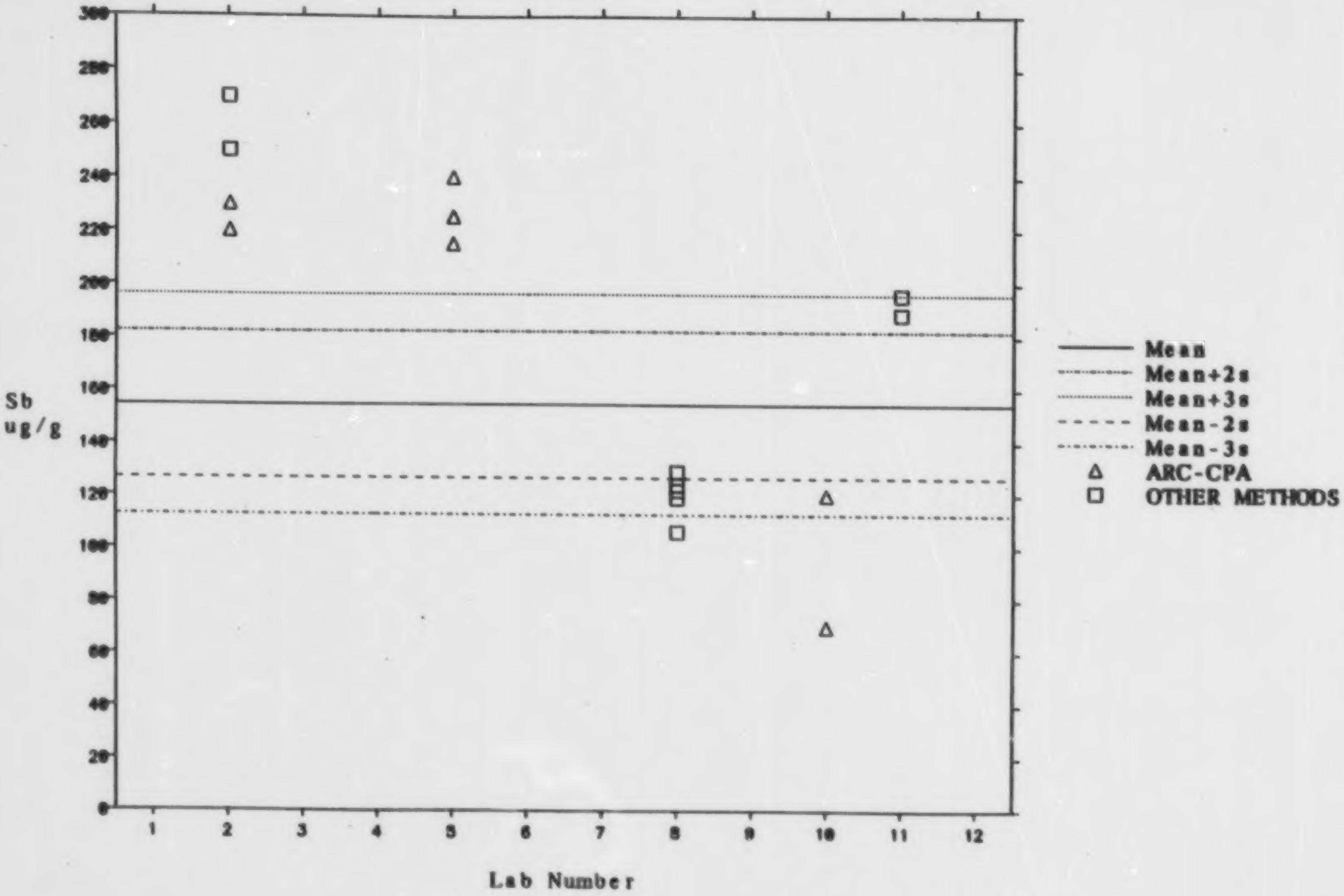


Table 16. Statistical Parameters for Sb (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	5.000	6.000	7.0	6.00	0	6.000
Sum	4.440	16.000	590.0	25.30		15.800
Mean	0.888	2.670	84.3	4.21		2.630
SEM	0.234	0.450	13.3	2.59		1.040
Median	0.800	3.040	84.0	1.59		2.730
Variance	0.273	1.210	1240.0	40.20		6.470
StDev	0.523	1.100	35.3	6.34		2.540
Maximum	1.700	3.600	120.0	17.00		5.300
Minimum	0.291	0.547	13.0	0.80		0.069
Range	1.410	3.050	107.0	16.20		5.230
Skewness	0.900	-1.890	-1.5	2.33		-0.009
Kurtosis	1.470	3.860	3.1	5.52		-3.160
CV(%)	58.800	41.300	41.9	151.00		96.800

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 154. Analysis of Sb in Sludge A

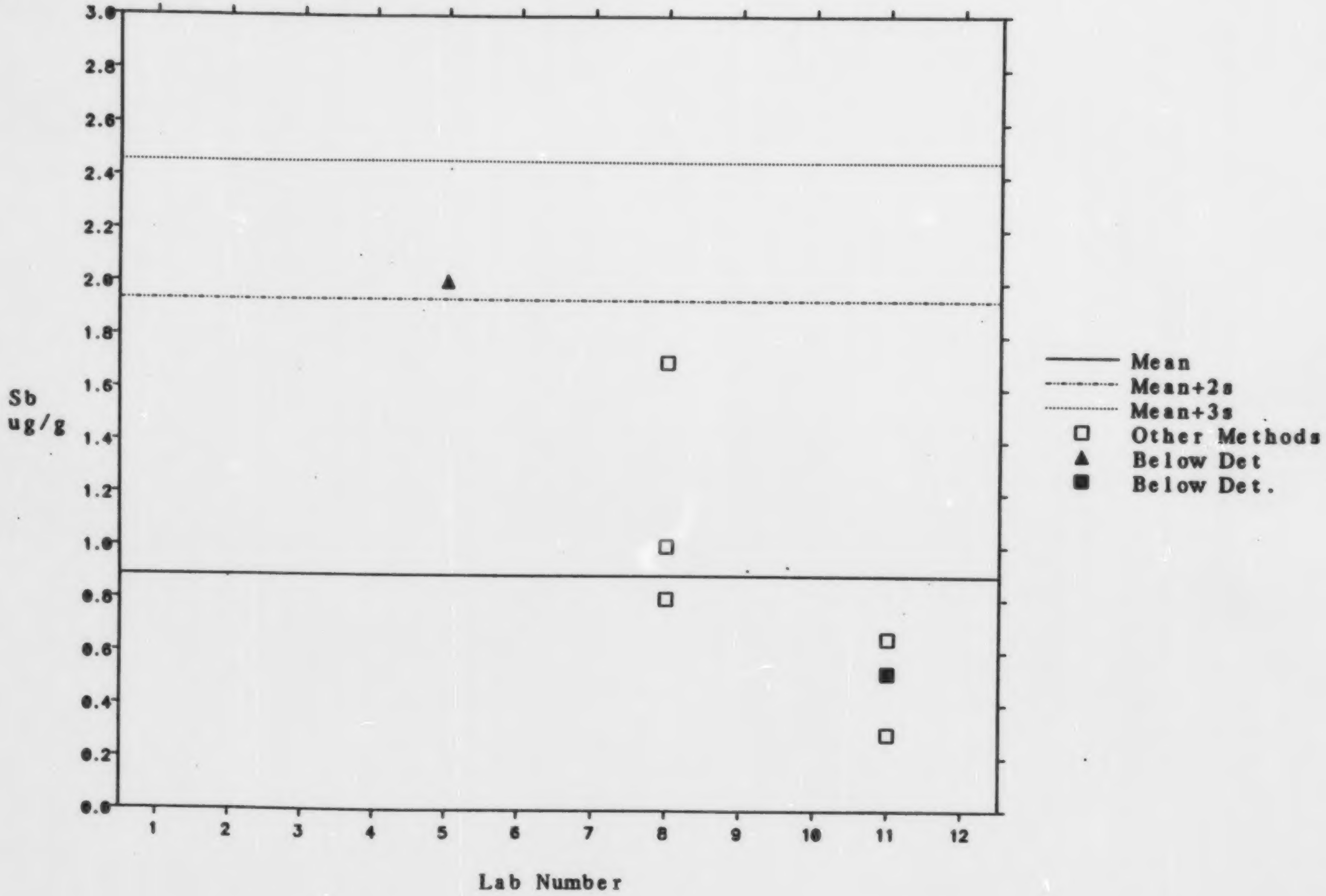


Figure 155. Analysis of Sb in Sludge B

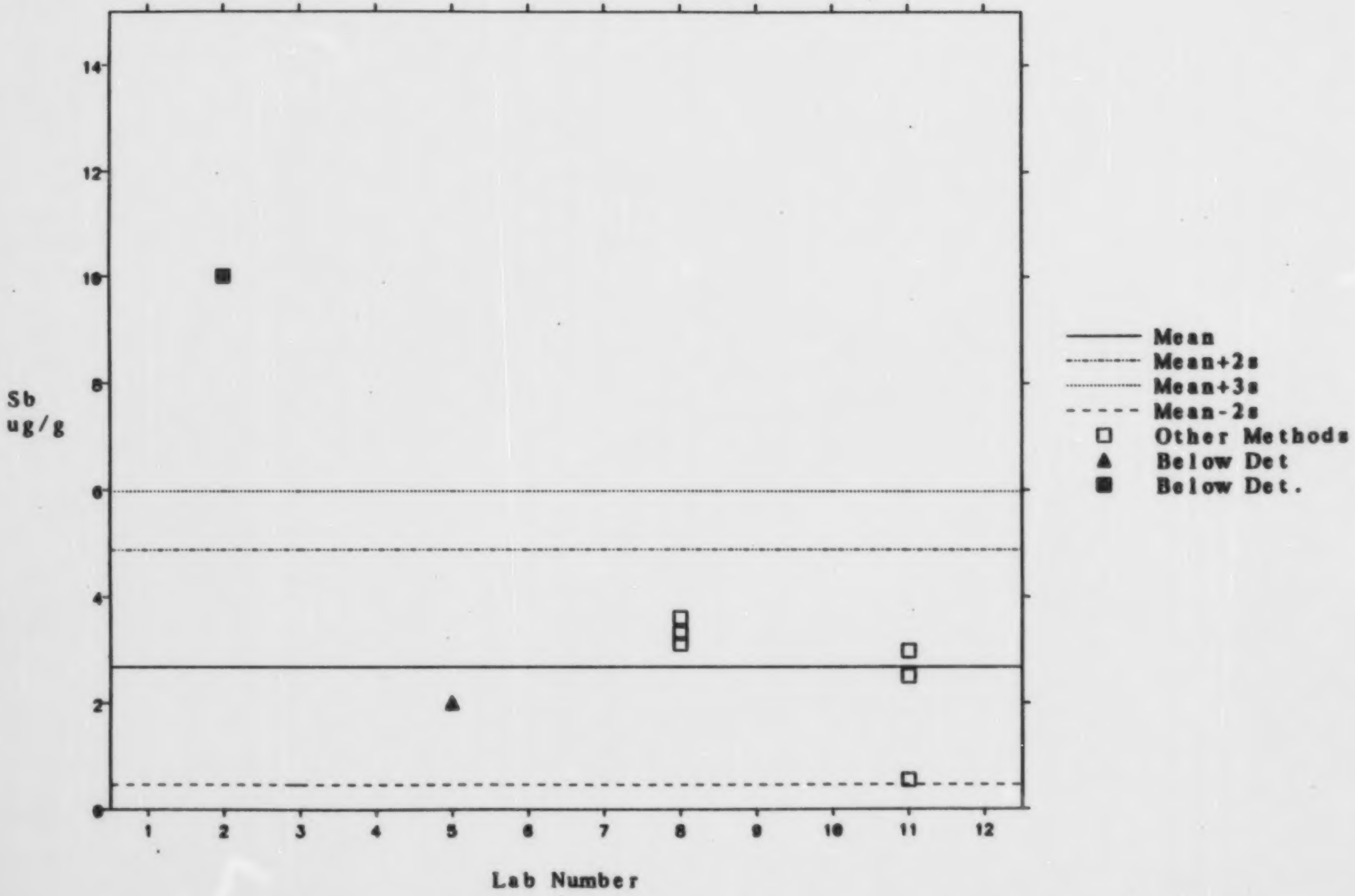


Figure 156. Analysis of Sb in Sludge C

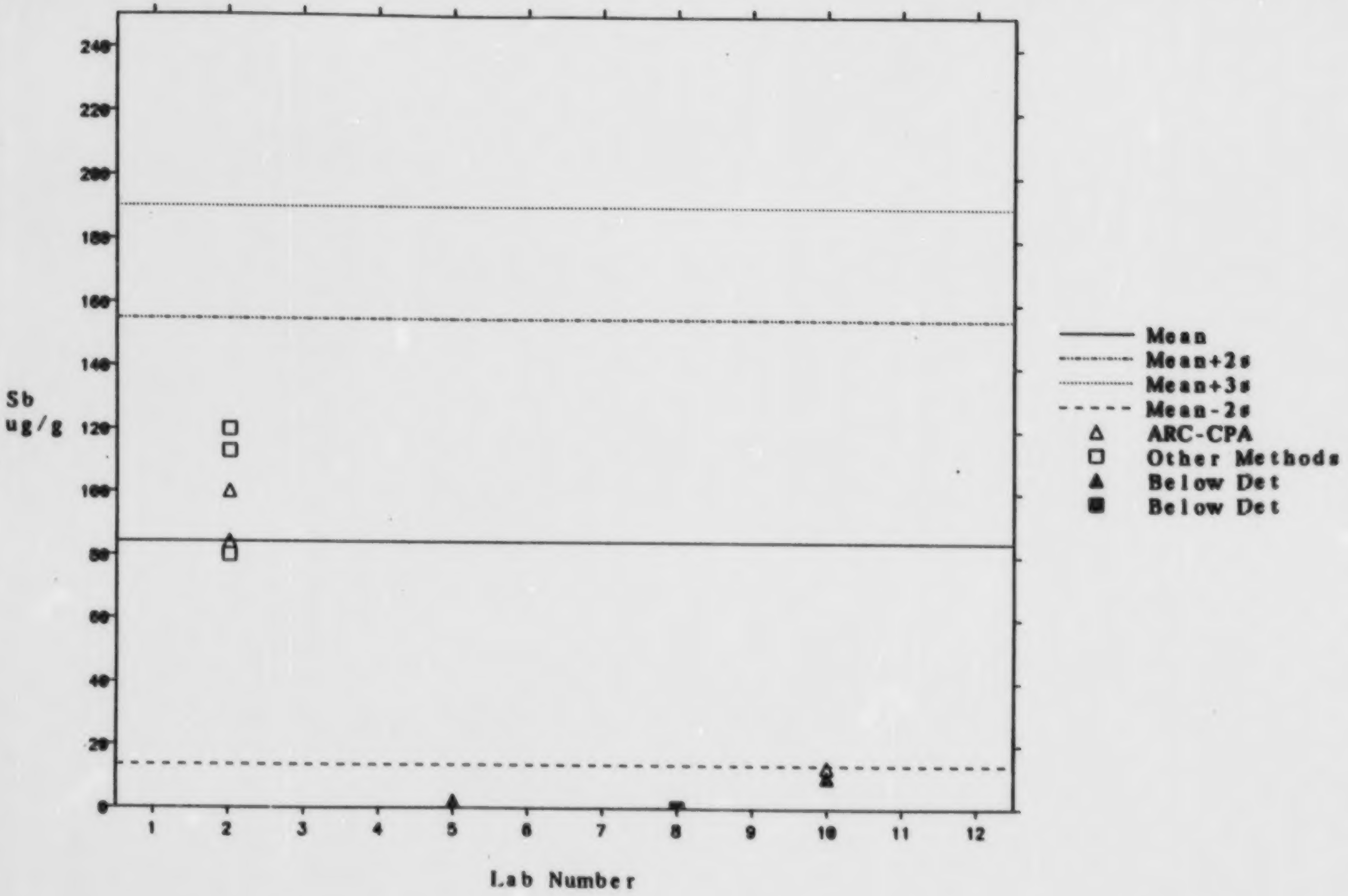


Figure 157. Analysis of Sb in Sludge D

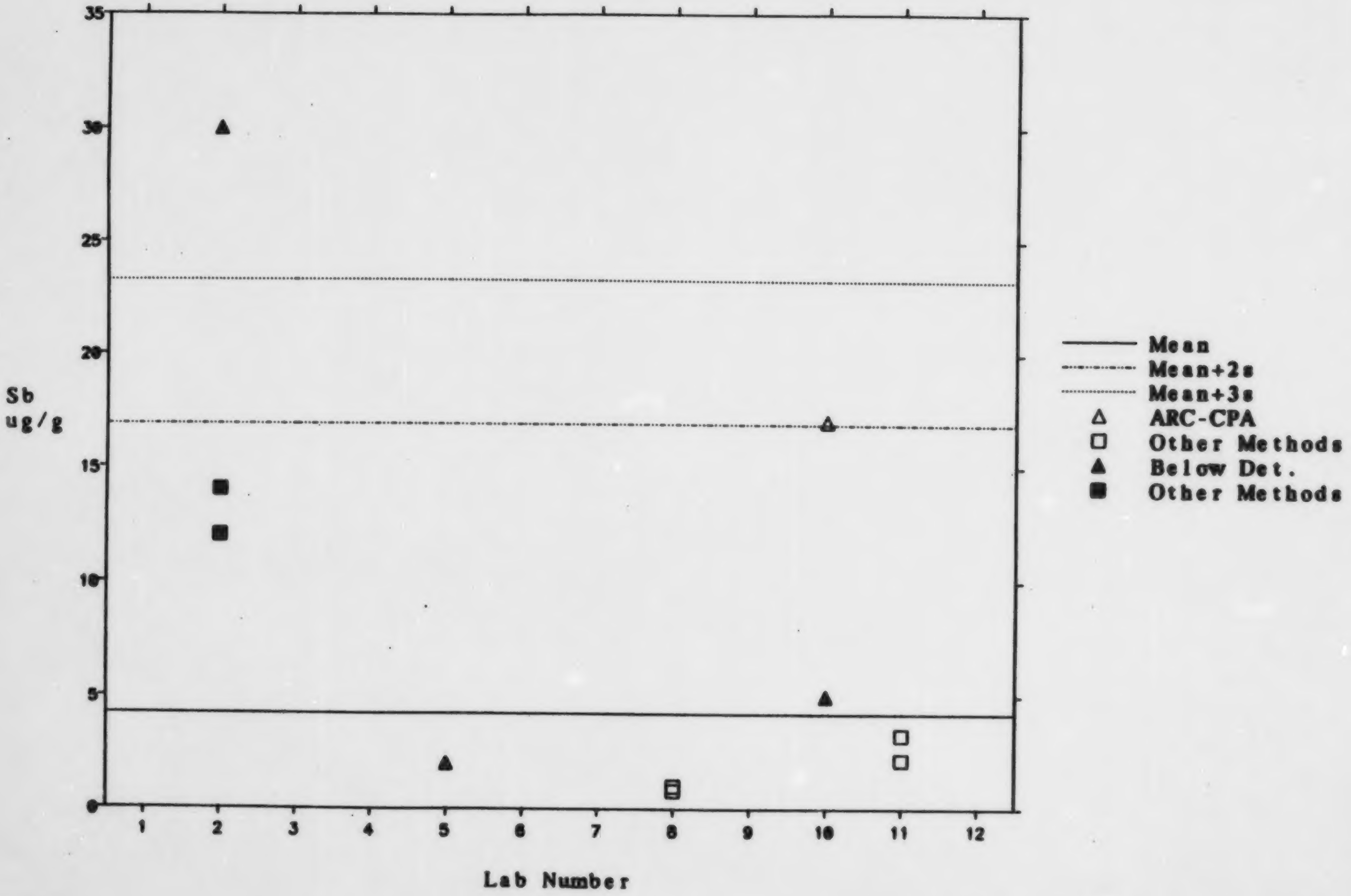


Figure 158. Analysis of Sb in Sludge E

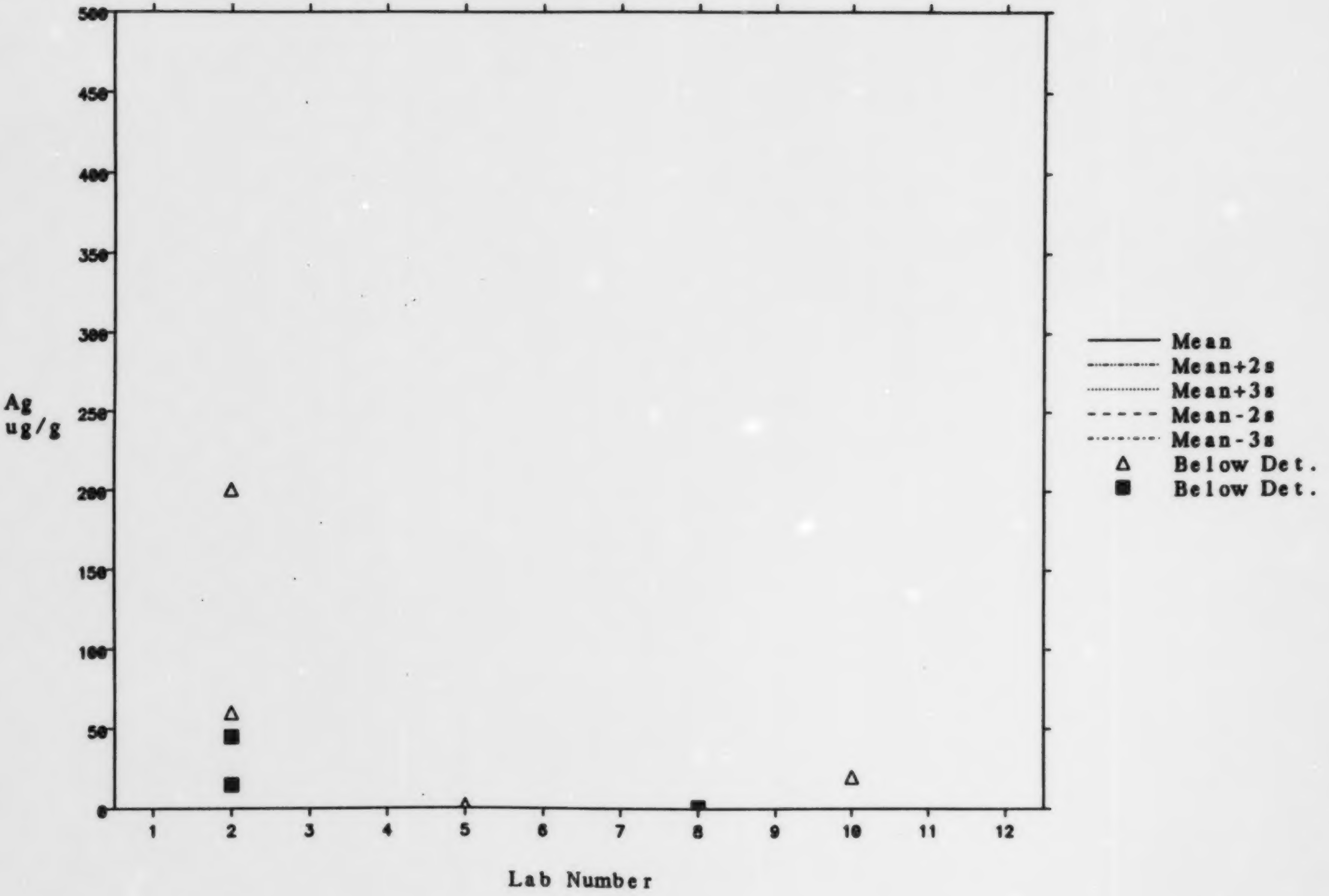
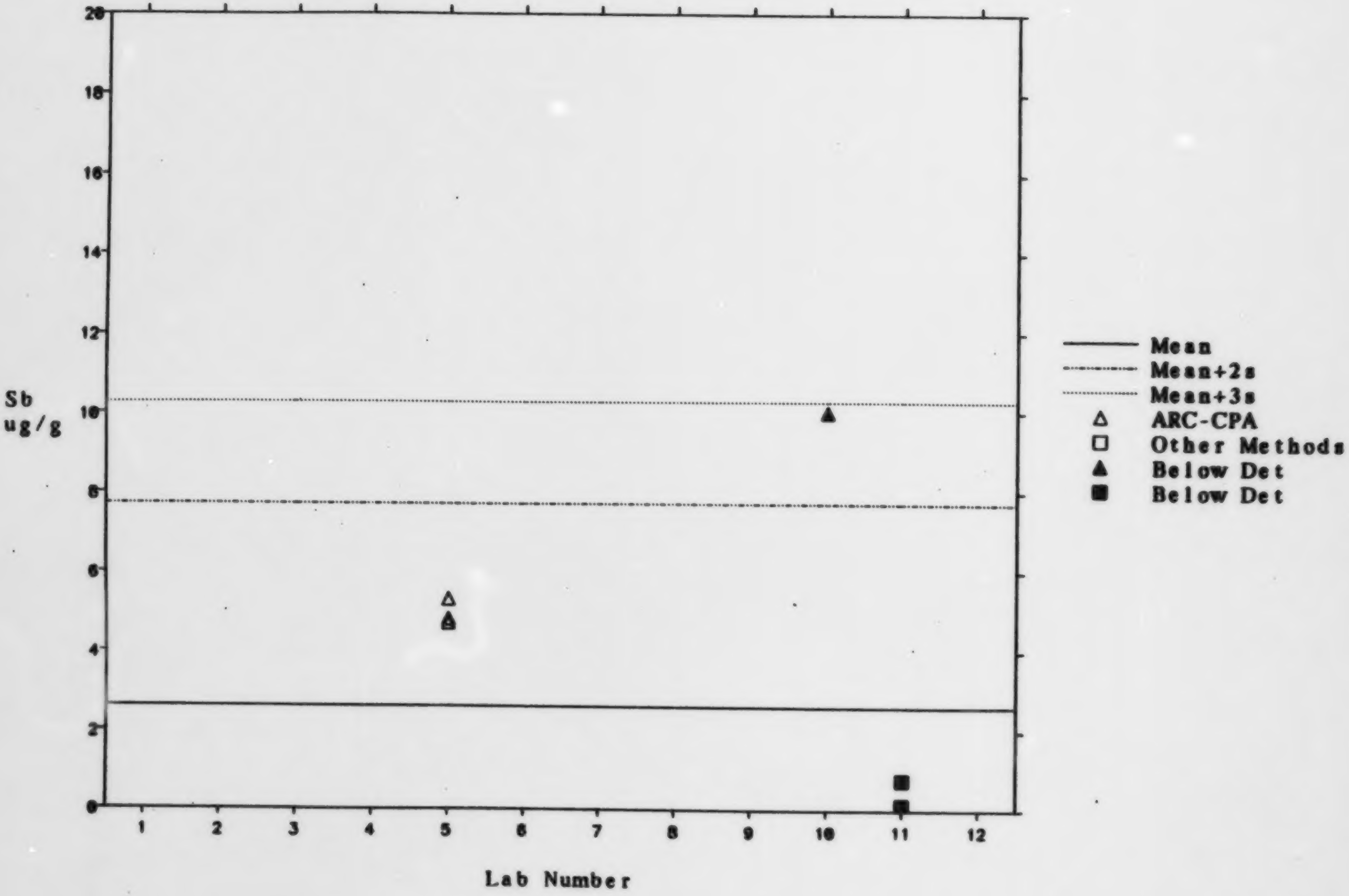


Figure 159. Analaysis of Sb in Sludge F



Figures 154 to 159 indicate that all the reported Sb data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods. The detection limits for Sb in the sludges varied widely between labs, with the range as high as one order of magnitude.

The Sb data were not many and that reflected on the agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed.

4.1.1.16 Se

Two labs reported Se in the ICP standard (Figure 160), both were lower than the low control limit. Four labs reported Se for the digestate 3 and two labs for the digestate B samples (Figures 161 and 162). Digestate 3 data (Figure 161) were outside the control limits except for one lab which was near the mean value. The two labs reporting digestate 4 data were widely separated with one near the mean and the other lower than the low control limit (Figure 162). No Se was reported by the labs in either Standard A or Standard B samples.

The statistical data for Se in sludges A to F are shown in Table 17, and their control charts in Figures 163 to 168. Table 17 shows that results (above detection) were reported for all sludges, with number of analyses varying from 3 to 12 samples. The mean Se values ranged from 0.05 mg/kg for sludge A to 0.28 for sludge C. The coefficient of variation of the sludges varied from 28 to 97% and it seems to be inversely related to the number of reported results.

Figures 163 to 168 indicate that all the reported Se data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods. The Sludge Se detection limits reported varied by more than an order of magnitude.

The scarceness of the Se data makes it difficult to interpret this element's recovery and performance between labs. The limited data shows some agreement between the different labs and for the different methodologies used. The variability seems to be dependent on the number of analyses performed, with lower CV attained when more samples are analyzed.

Figure 160. Mean and Confidence Intervals for Se in an ICP Standard

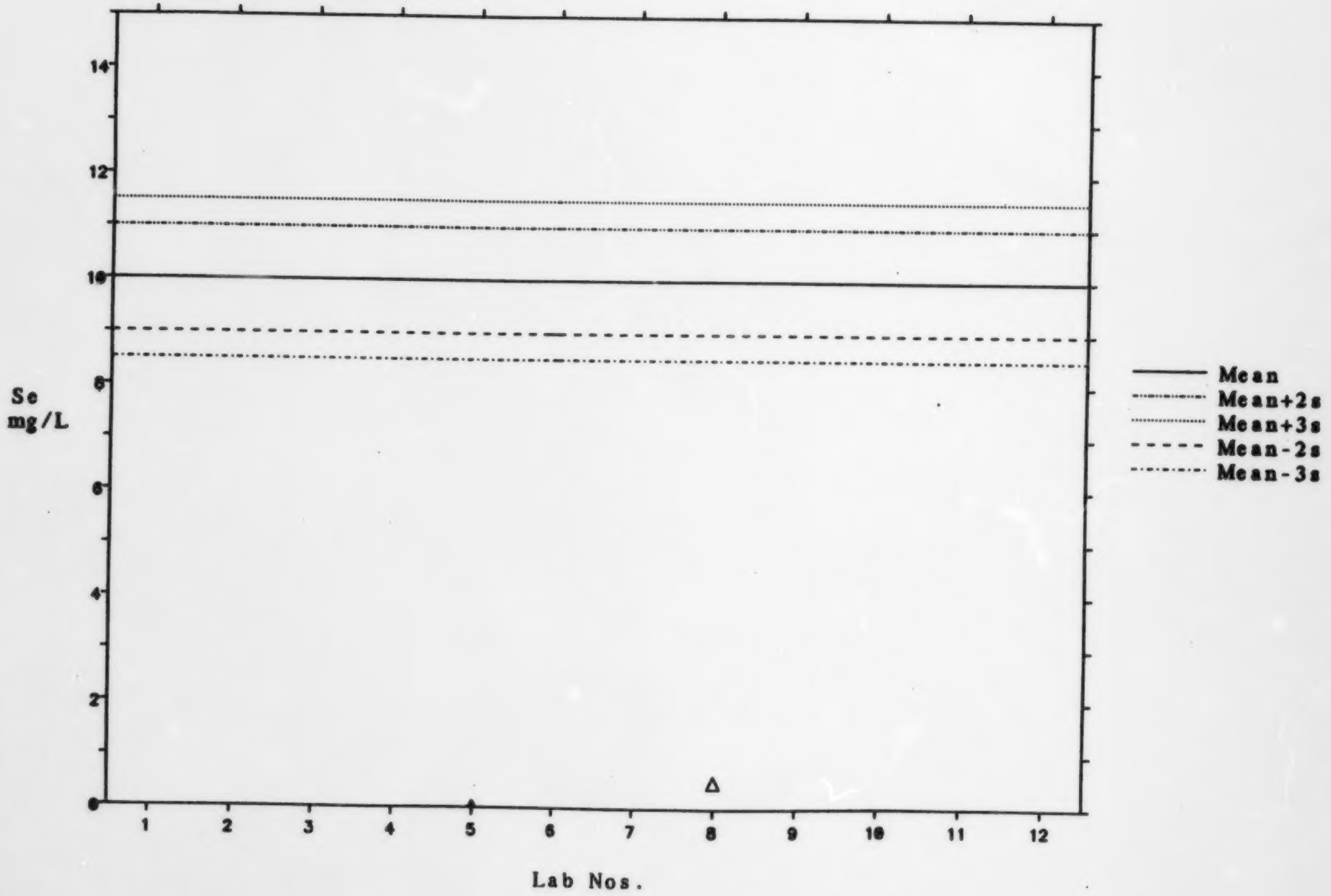


Figure 161. Mean and Confidence Intervals for Se in Digestate 3

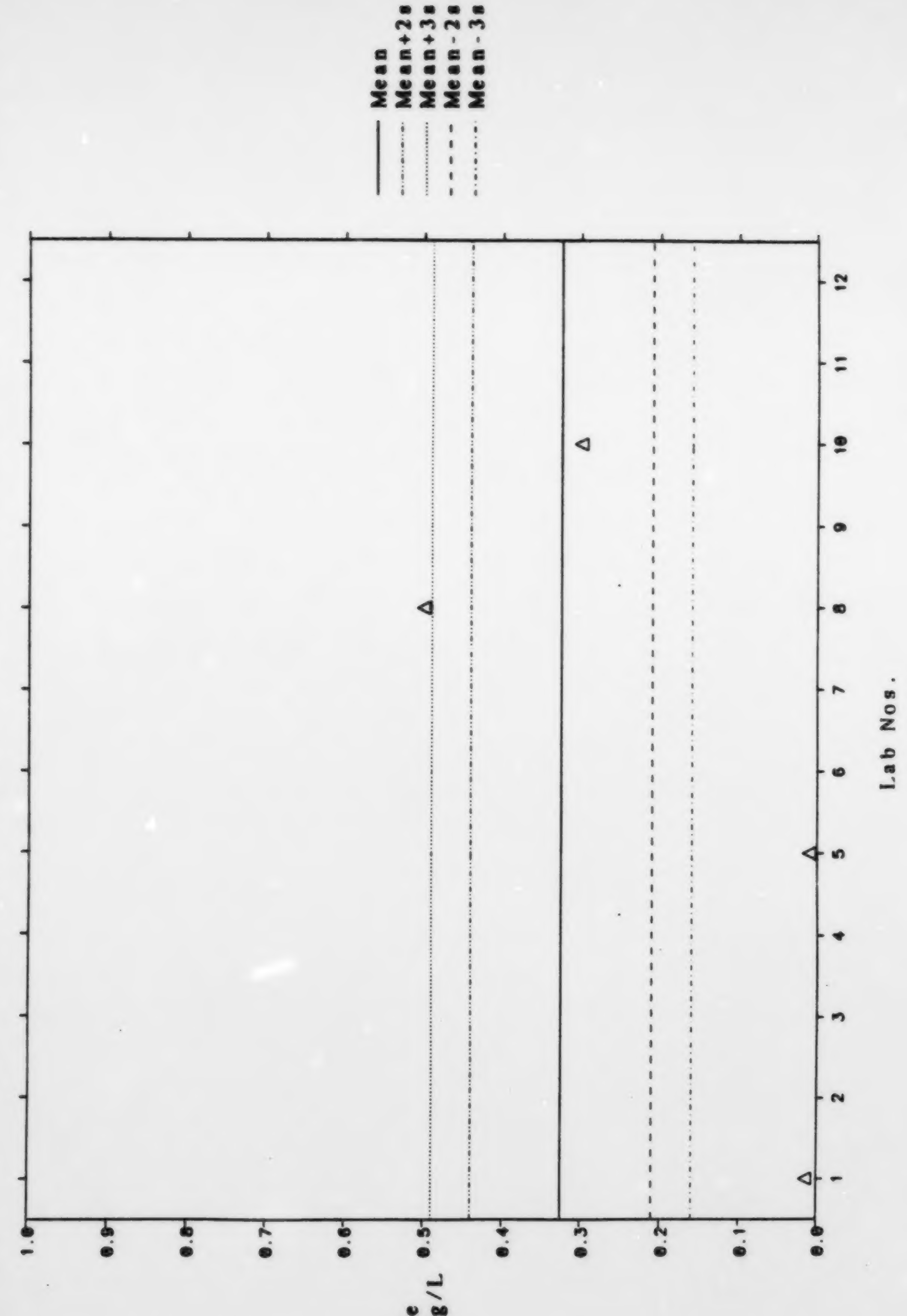


Figure 162. Mean and Confidence Intervals for Se in Digestate 4

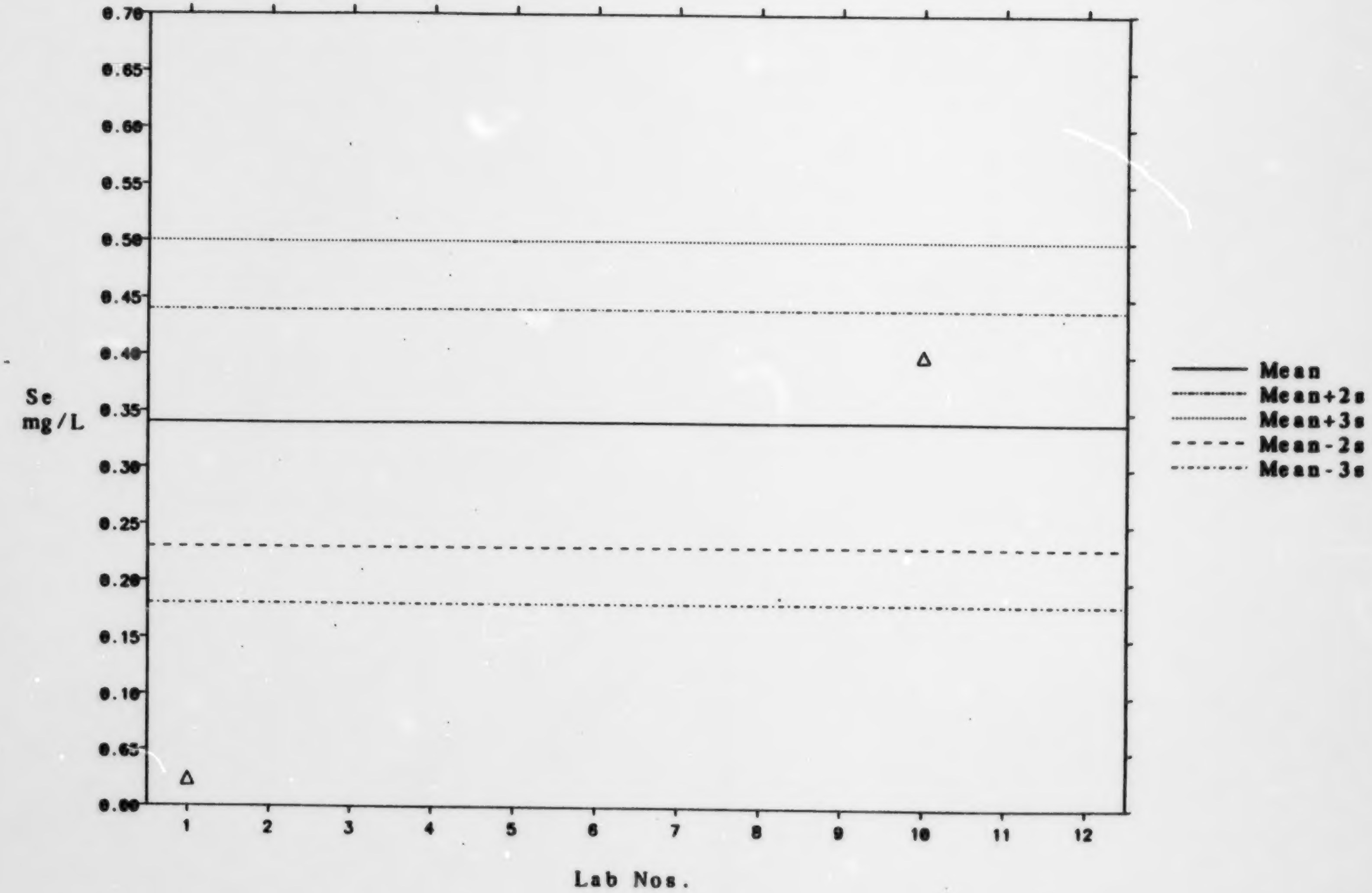


Table 17. Statistical Parameters for Se (mg/Kg) in Sludges A-F.

Statistic	Sludge A	Sludge B	Sludge C	Sludge D	Sludge E	Sludge F
Count	6	6.000	12.000	6.000	3.000	6
Sum	3e-01	1.180	3.360	0.550	0.500	1
Mean	5e-02	0.197	0.280	0.092	0.167	2e-01
SEM	1e-02	0.032	0.034	0.015	0.094	2e-02
Median	5e-02	0.195	0.310	0.085	0.110	2e-01
Variance	9e-04	0.006	0.014	0.001	0.026	2e-03
StDev	3e-02	0.077	0.117	0.036	0.163	5e-02
Maximum	8e-02	0.280	0.390	0.140	0.350	2e-01
Minimum	2e-02	0.120	0.090	0.060	0.040	1e-01
Range	6e-02	0.160	0.300	0.080	0.310	9e-02
Skewness	1e-16	0.029	-0.782	0.299	1.380	-2e-16
Kurtosis	-3	-3.190	-1.020	-2.410		-3
CV(%)	59	39.200	41.700	39.300	97.500	28

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 163. Analysis of Se in Waste A

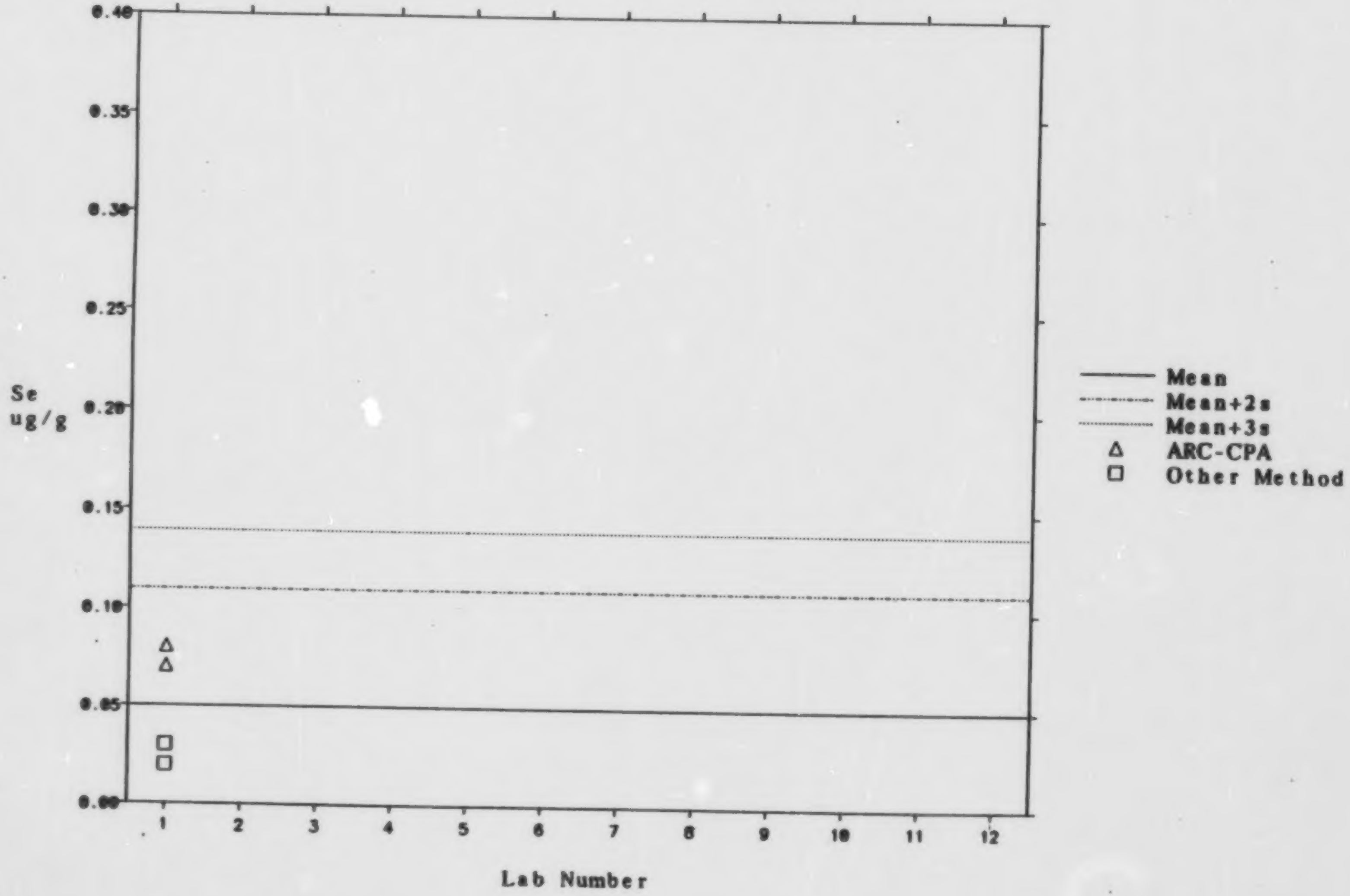


Figure 164. Analysis of Se in Sludge B

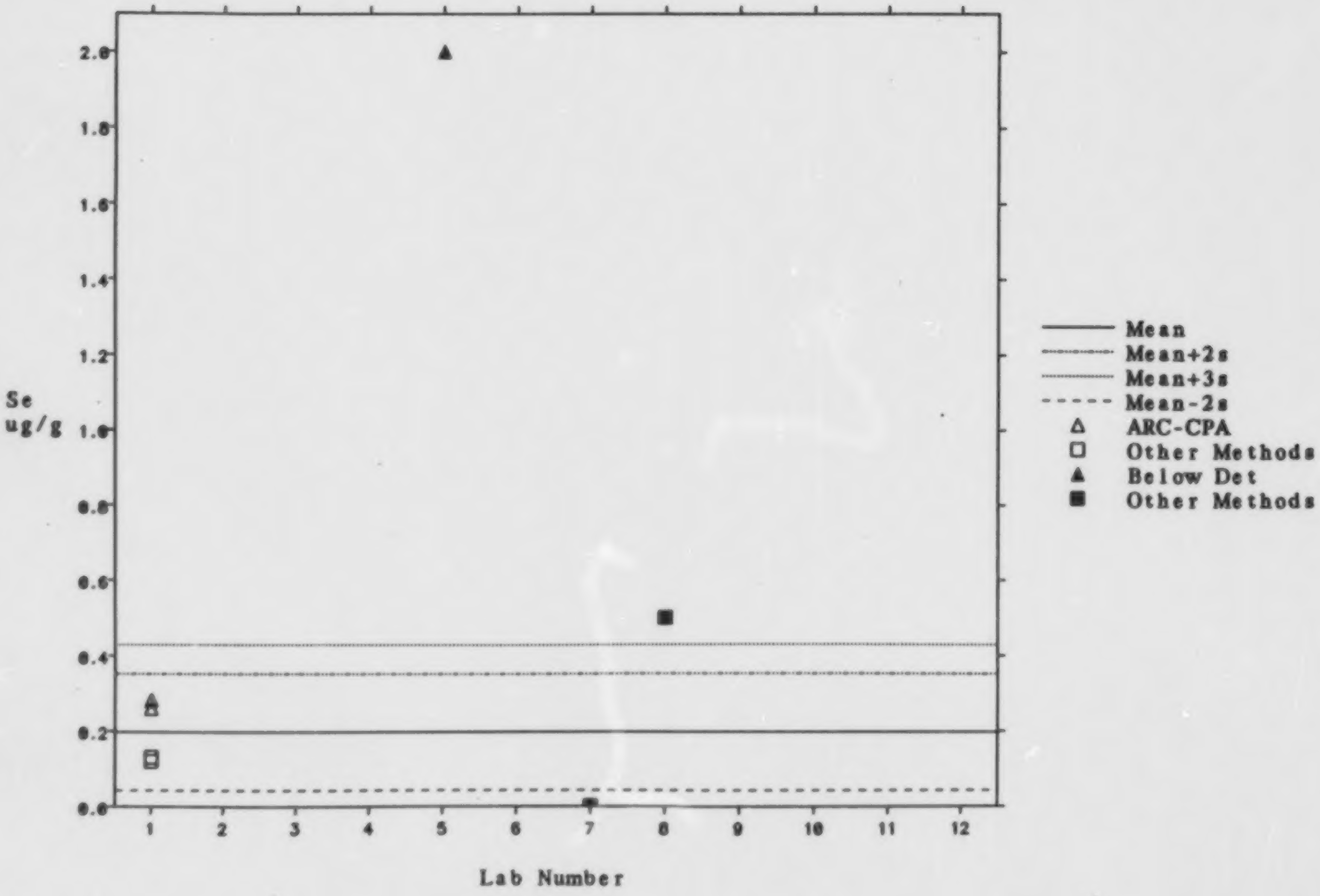


Figure 165. Analysis of Se in Sludge C

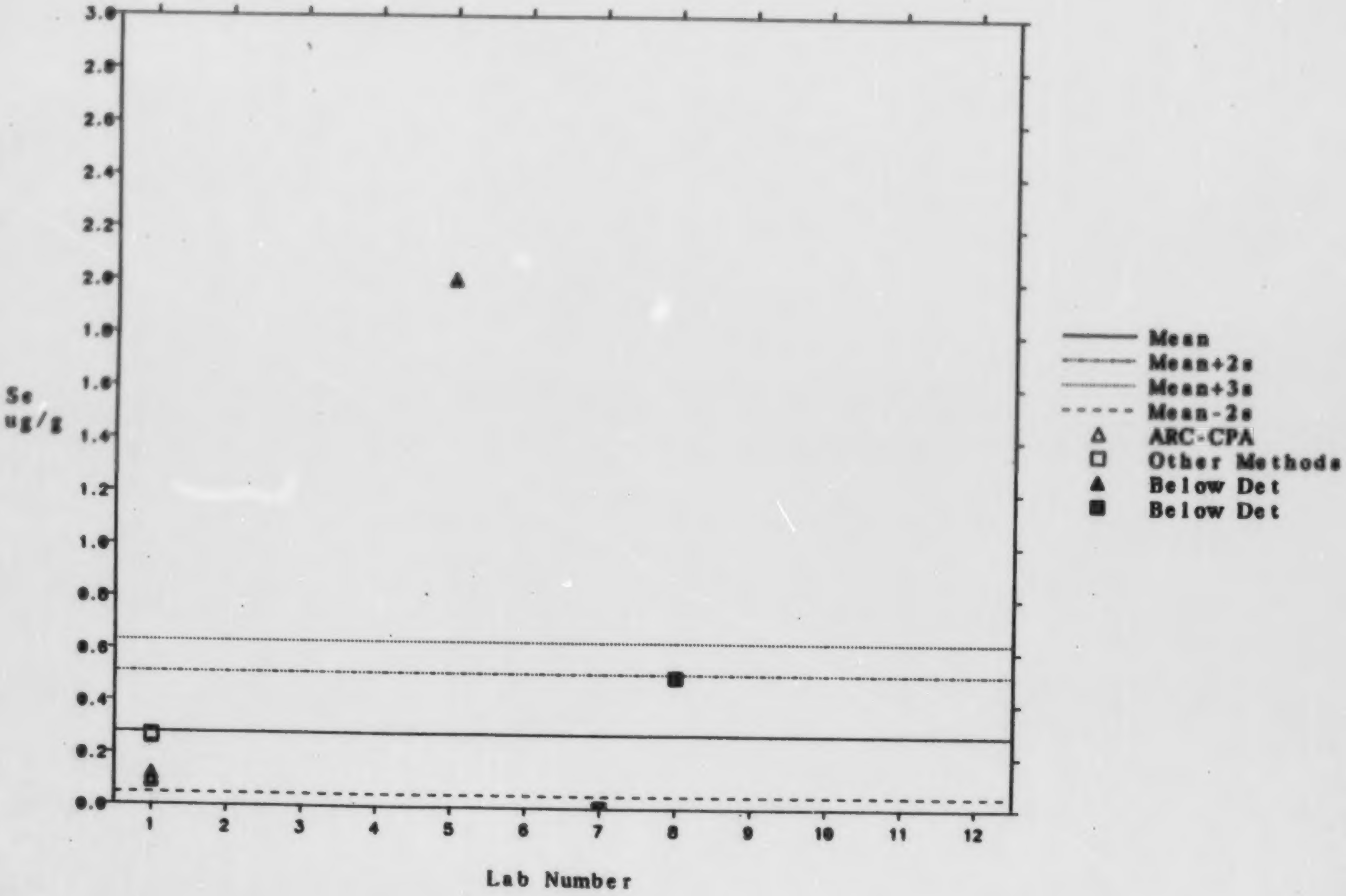


Figure 166. Analysis of Se in Sludge D

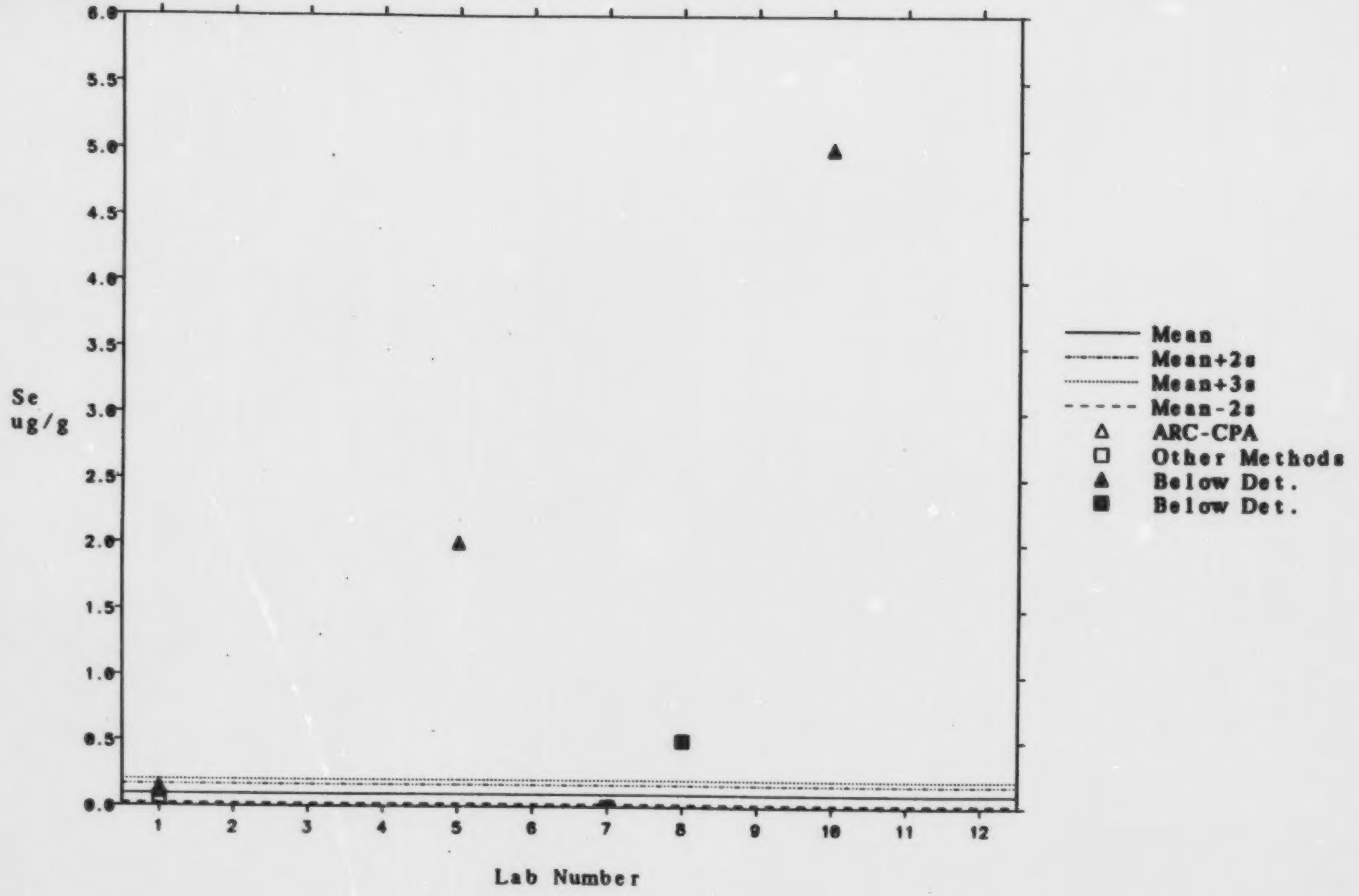


Figure 167. Analysis of Se in Sludge E

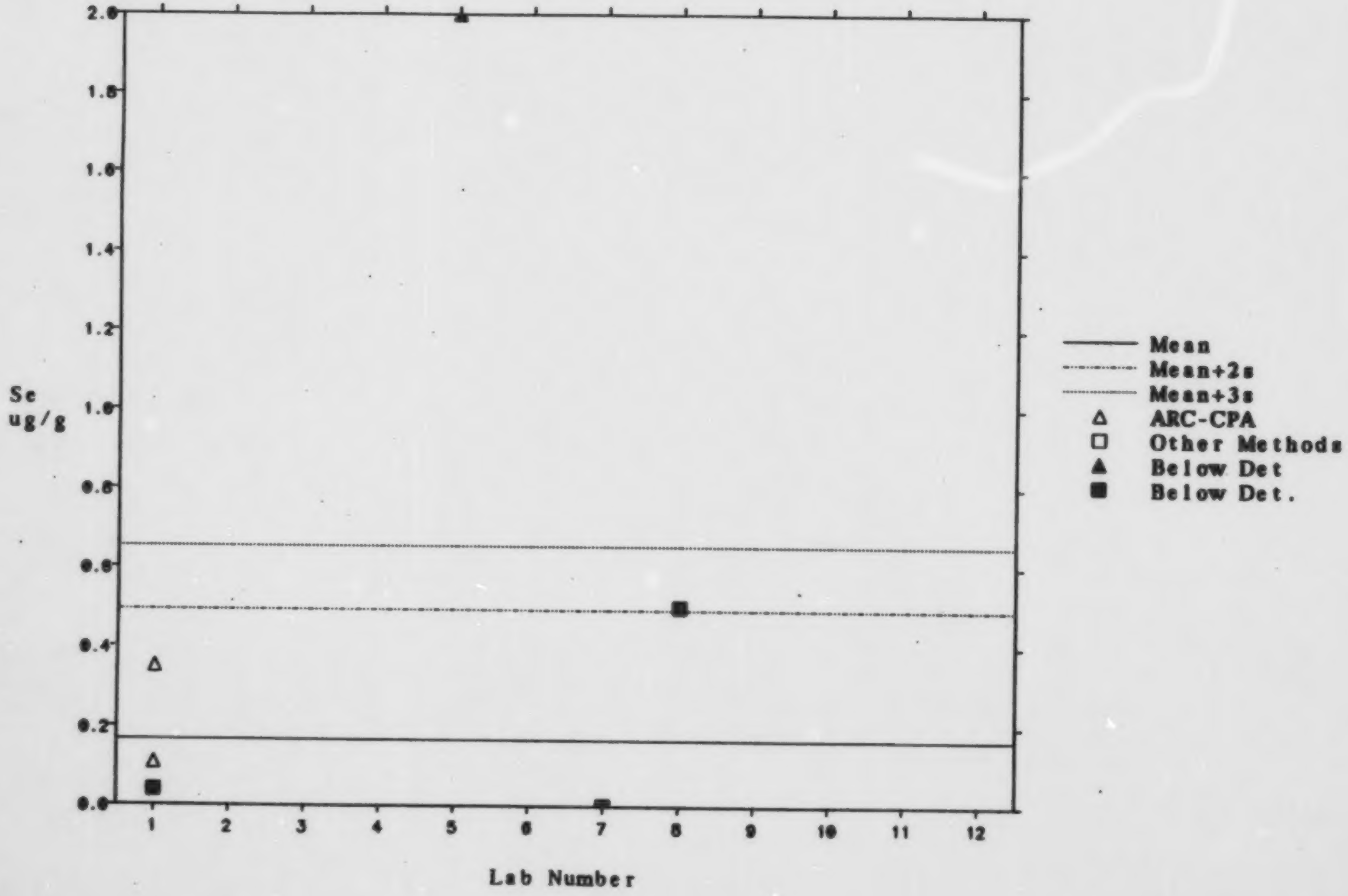
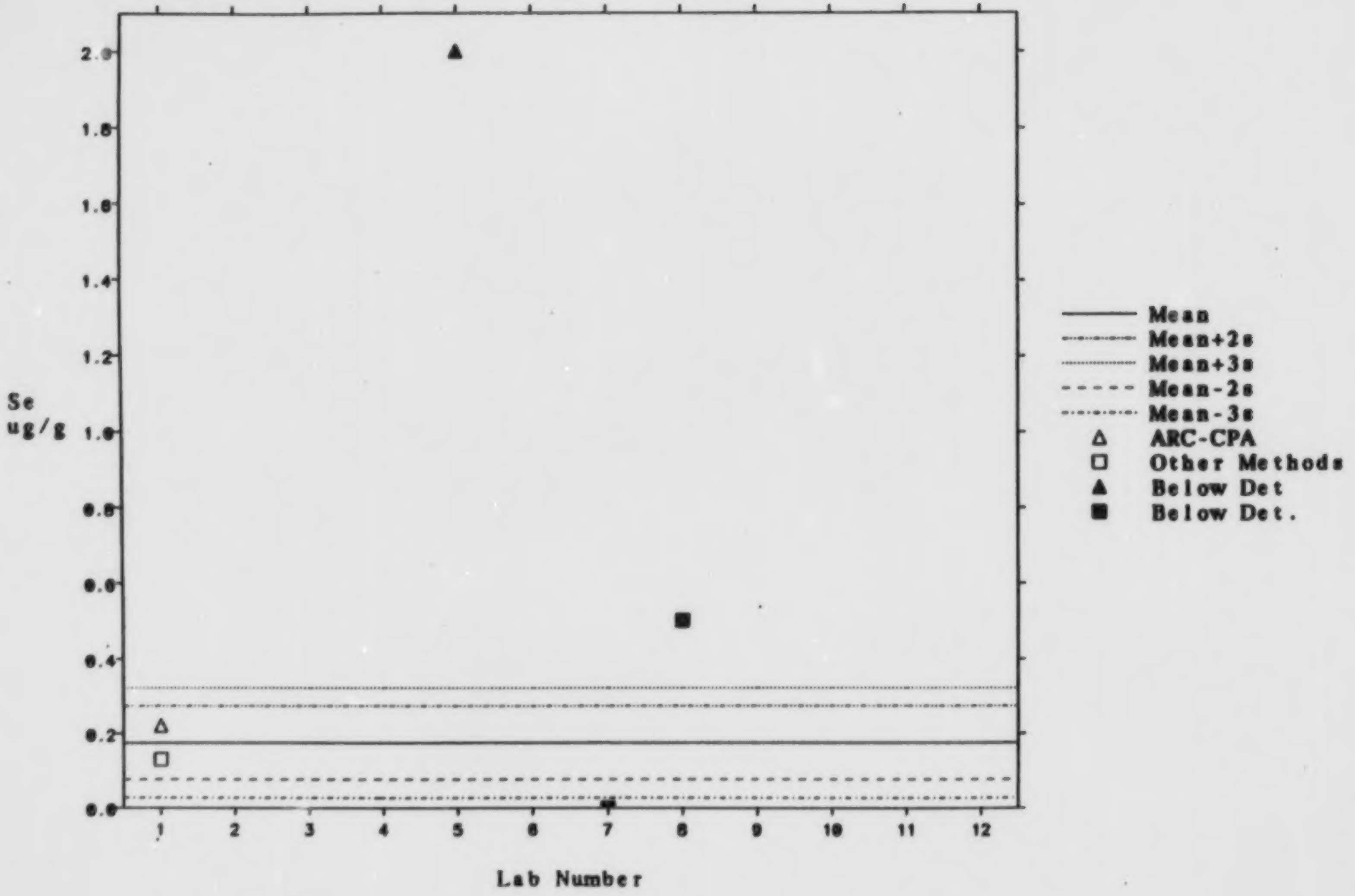


Figure 168. Analysis of Se in Sludge F



4.1.1.17 Tl

Two labs reported Tl in the ICP standard (Figure 169), both were much lower than the low control limit. Three labs reported Tl for the digestate 3 and two labs for the digestate 4 samples (Figures 170 and 171). Digestate 3 and 4 data (Figures 170 and 171) were all outside the control limits. Three labs reported Tl for the standard A and B samples (Figures 172 and 173). Figure 172 shows that 1 lab reported data within Standards A's control limits and the other 2 labs were outside those limits. Standard B values (Figure 173) show that 2 labs were completely within the control limits, with the other lab reporting some data both inside and outside the control limits. Figures 172 and 173 also show that the results of the ARC-CPA method were not different from those of other methods.

The statistical data for Tl in sludges A to F are shown in Table 18, and their control charts in Figures 174 to 176. Table 18 shows that results (above detection) were reported for 4 sludges, with number of analyses varying from 1 to 4 samples. The mean Tl values ranged from 1.2 mg/kg for sludge B to 15.6 for sludge D. The coefficient of variation of the sludges varied from 76 to 141% and it seems to be inversely related to the number of reported results.

Figures 174 to 176 indicate that all the reported Tl data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The interpretation of Tl performance suffers from lack of reported data as some of the elements already discussed. The small number of reported analyses makes it difficult to render any meaningful conclusions about the Tl recovery and lab performance.

4.1.1.18 V

Seven labs reported V in the ICP standard (Figure 177), all but two labs were within the warning limits. The two outlier labs were less than the lower control limit. Eight labs reported V for the digestate samples (Figures 178 and 179). Digestate 3 data (Figure 178) showed 7 labs

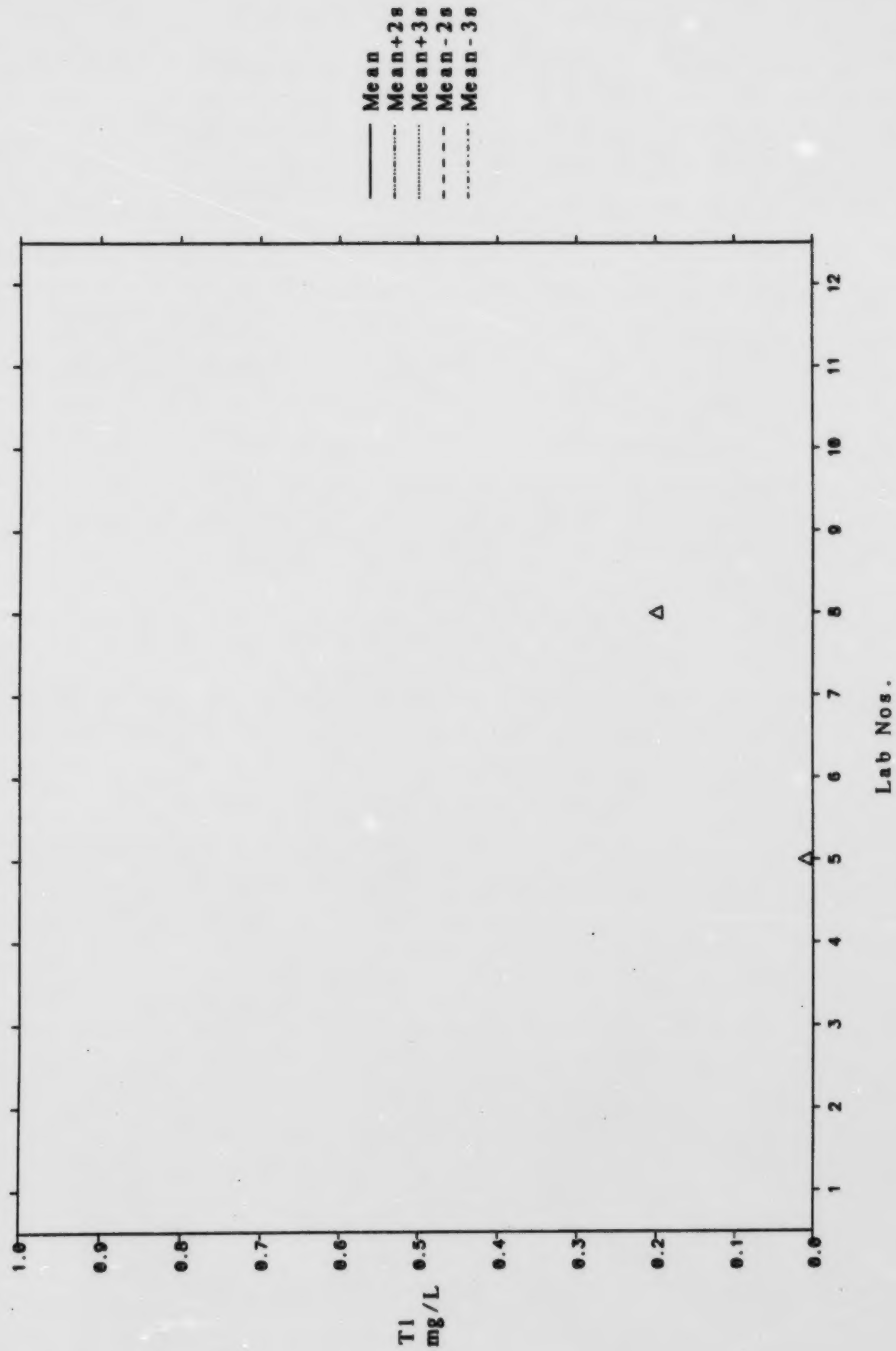


Figure 169. Mean and Confidence Intervals for Tl in an ICP Standard

Figure 170. Mean and Confidence Intervals for Tl in Digestate 3

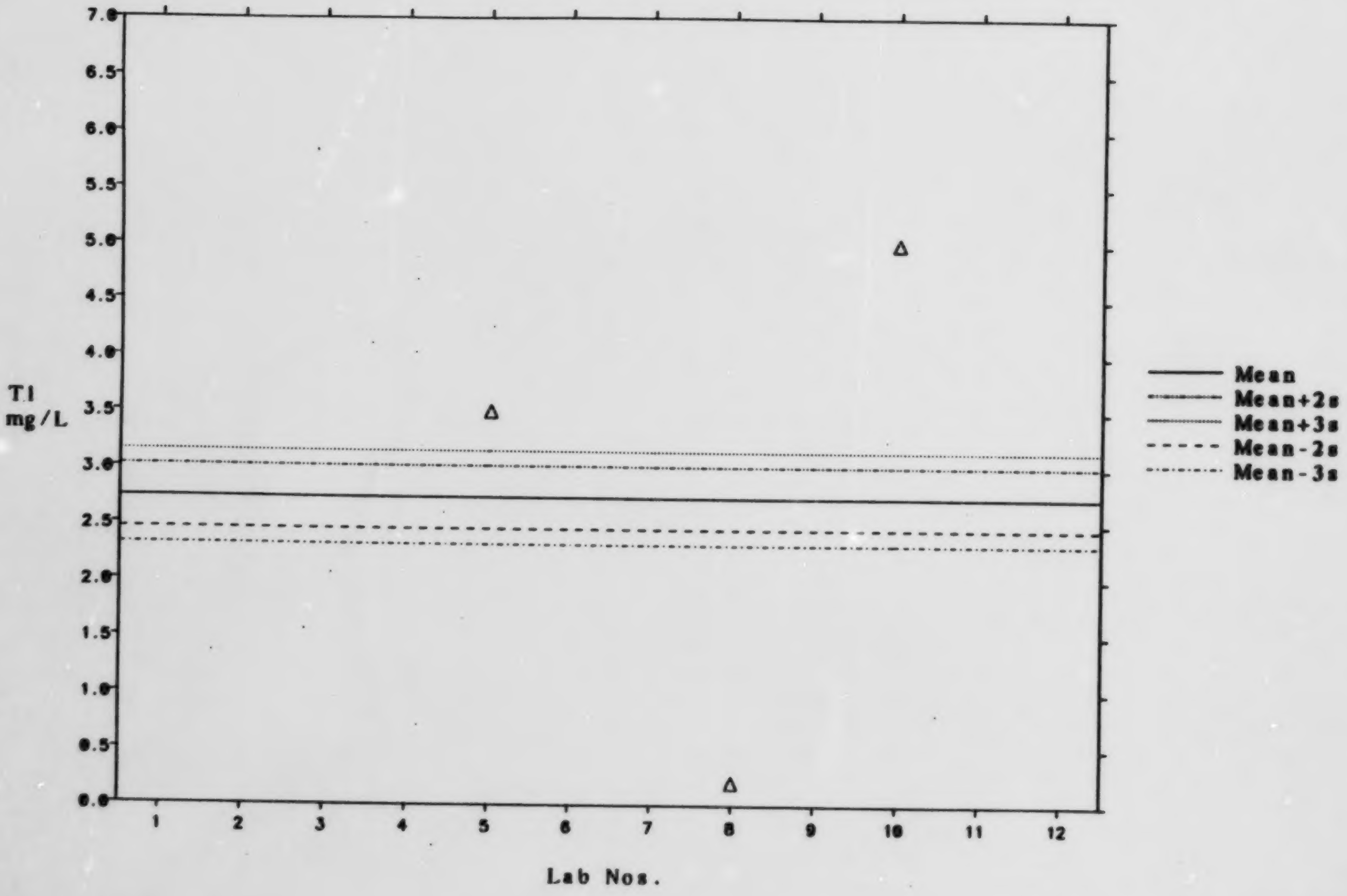


Figure 171. Mean and Confidence Intervals for Tl in Digestate 4

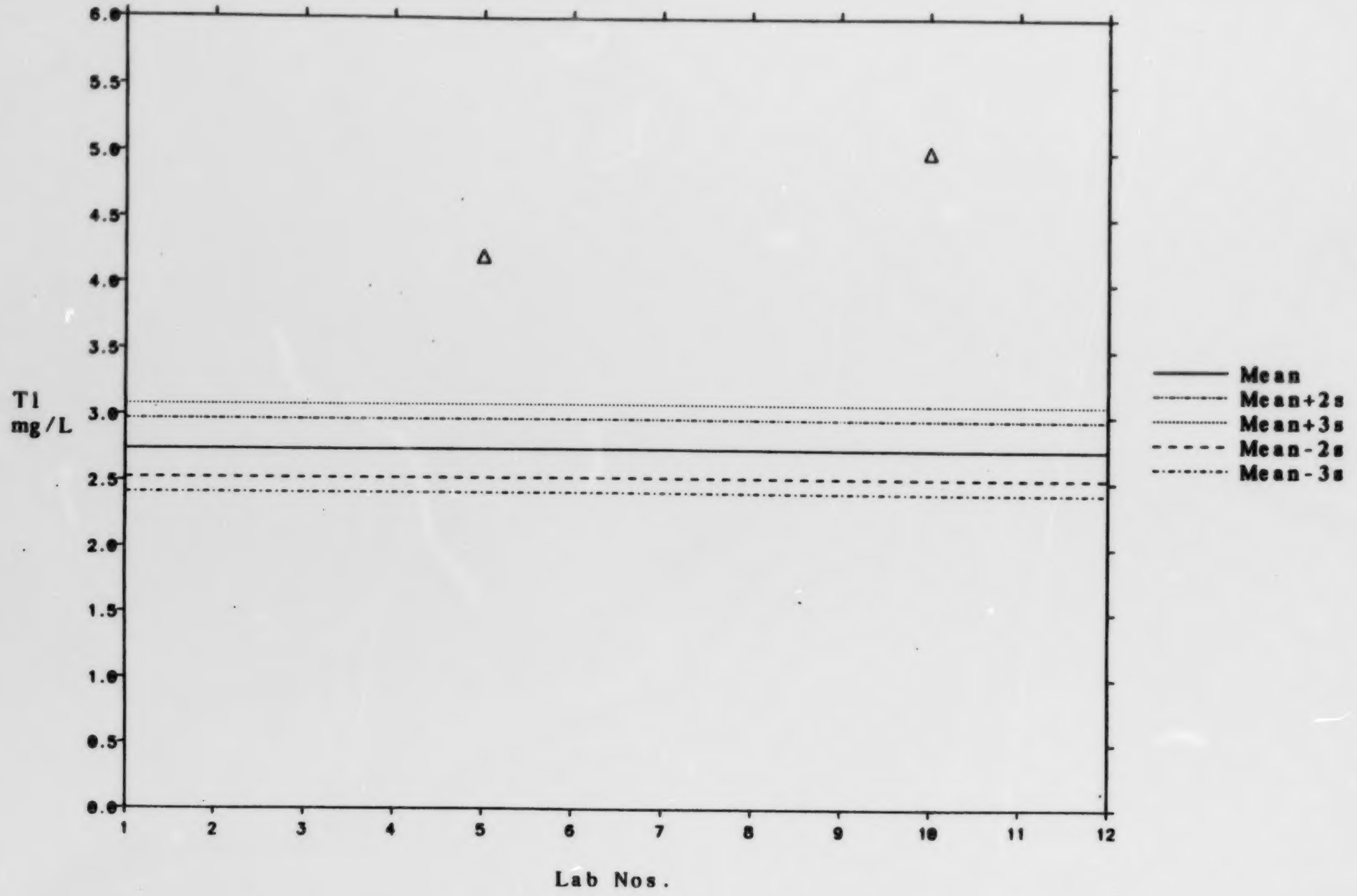


Figure 172. Mean and Confidence Intervals for Tl in Standard A

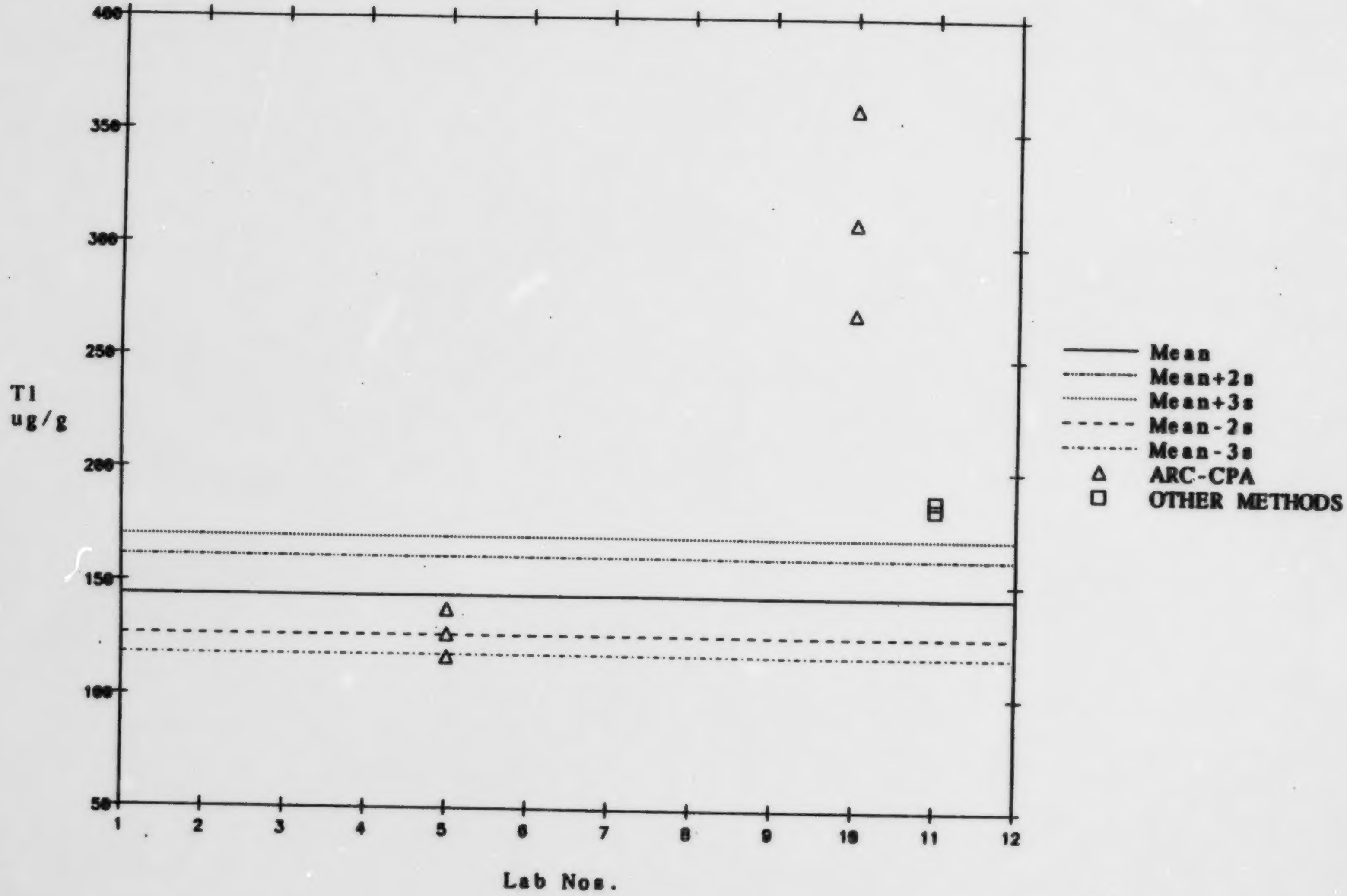


Figure 173. Mean and Confidence Intervals for Tl in Standard B

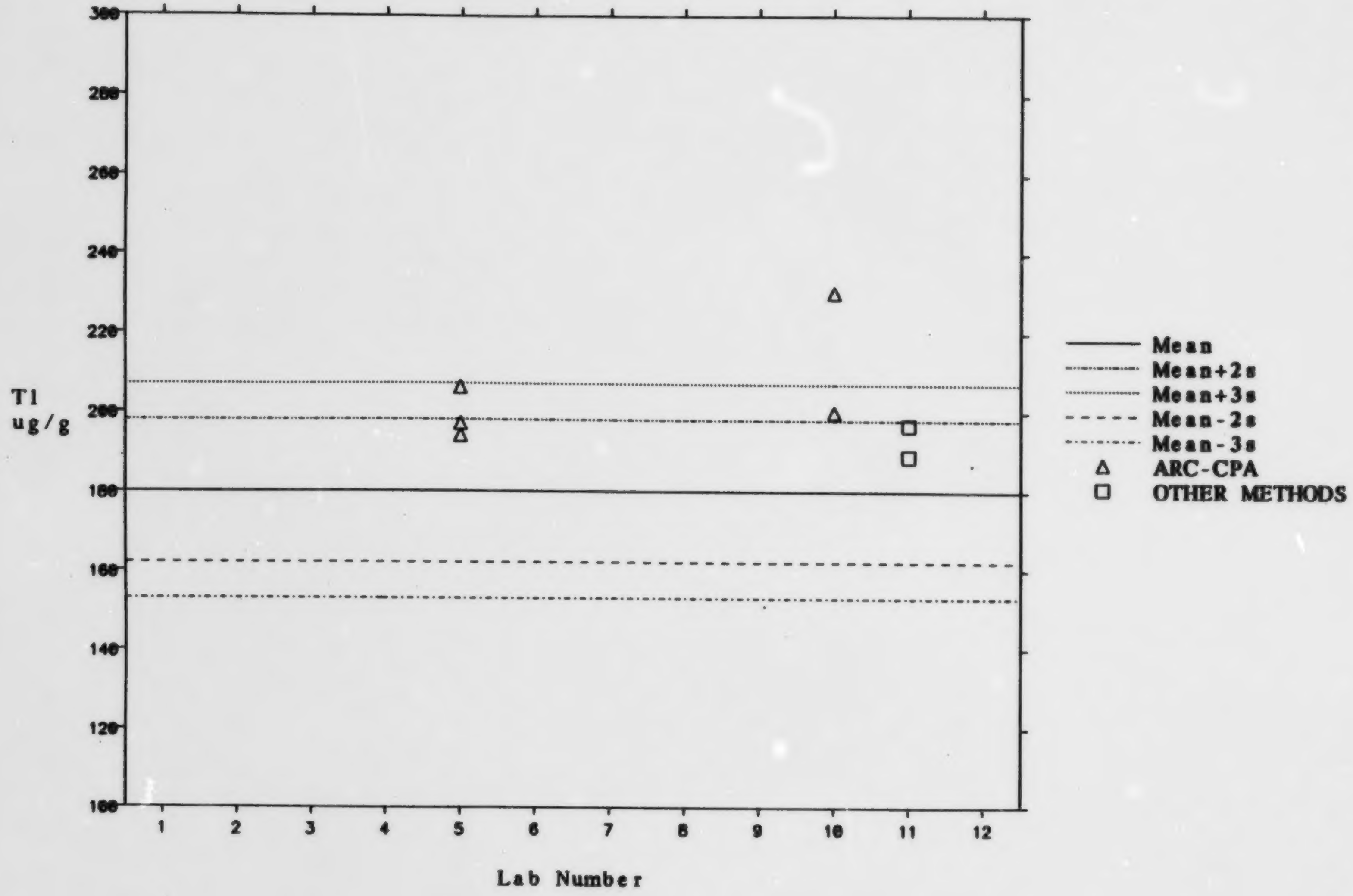


Table 18. Statistical Parameters for Tl (mg/kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	0	1.00	0	4.00	2.000	2.00
Sum		1.23		62.30	4.600	31.10
Mean		1.23		15.60	2.300	15.50
Sem				5.95	1.480	15.50
Median		1.23		18.00	2.300	15.50
Variance				142.00	4.370	478.00
Stdev				11.90	2.090	21.90
Maximum		1.23		26.00	3.780	31.00
Minimum		1.23		0.31	0.824	0.07
Range		0.00		25.70	2.960	30.90
Skewness				-0.74		
Kurtosis				-1.68		
CV(%)				76.50	90.800	141.00

Sem=standard error of mean; StDev= standard deviation;
C.V=coefficient of variability;

Figure 174. Analysis of Tl in Sludge D

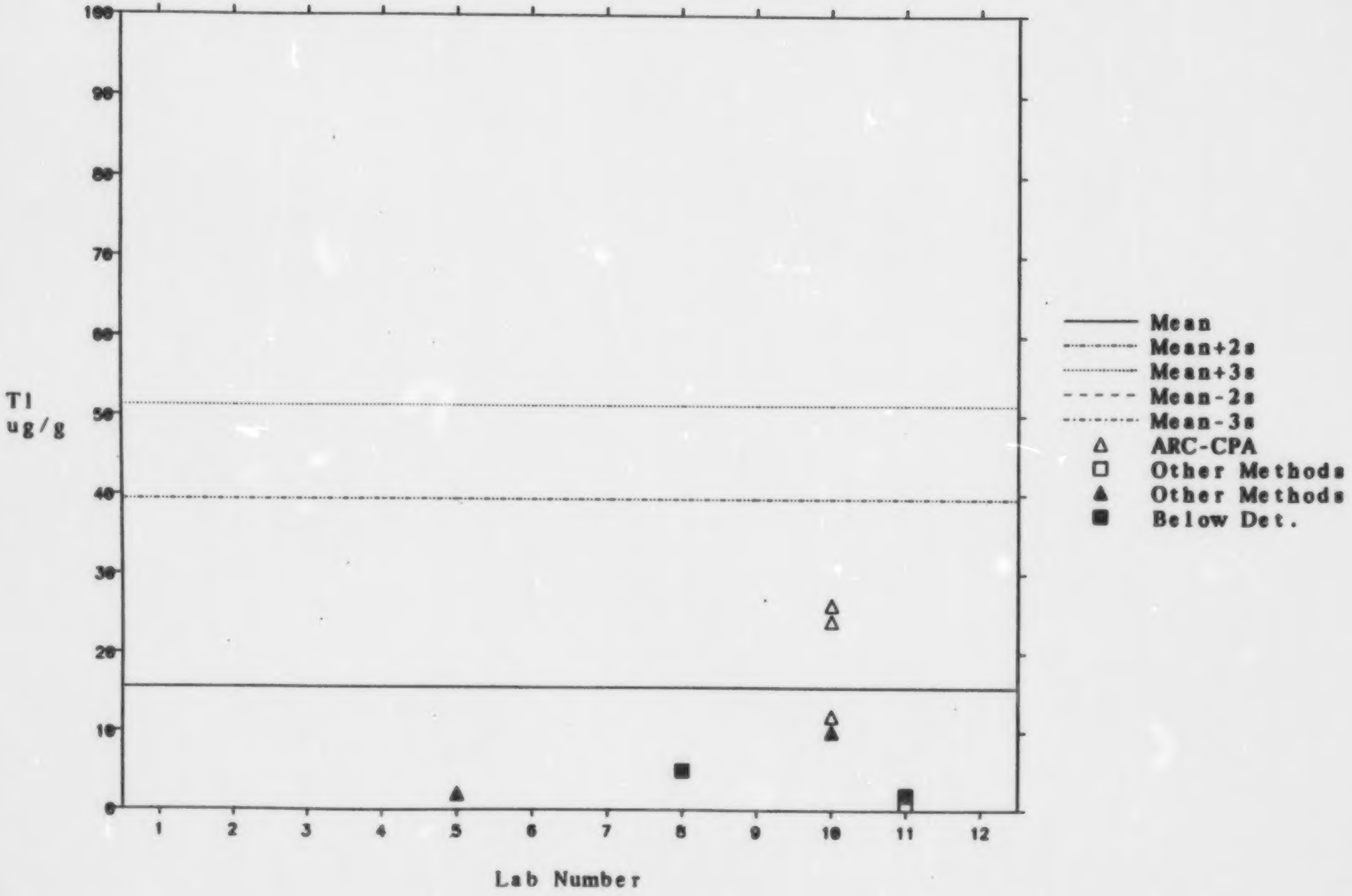


Figure 175. Analysis of Tl in Sludge E

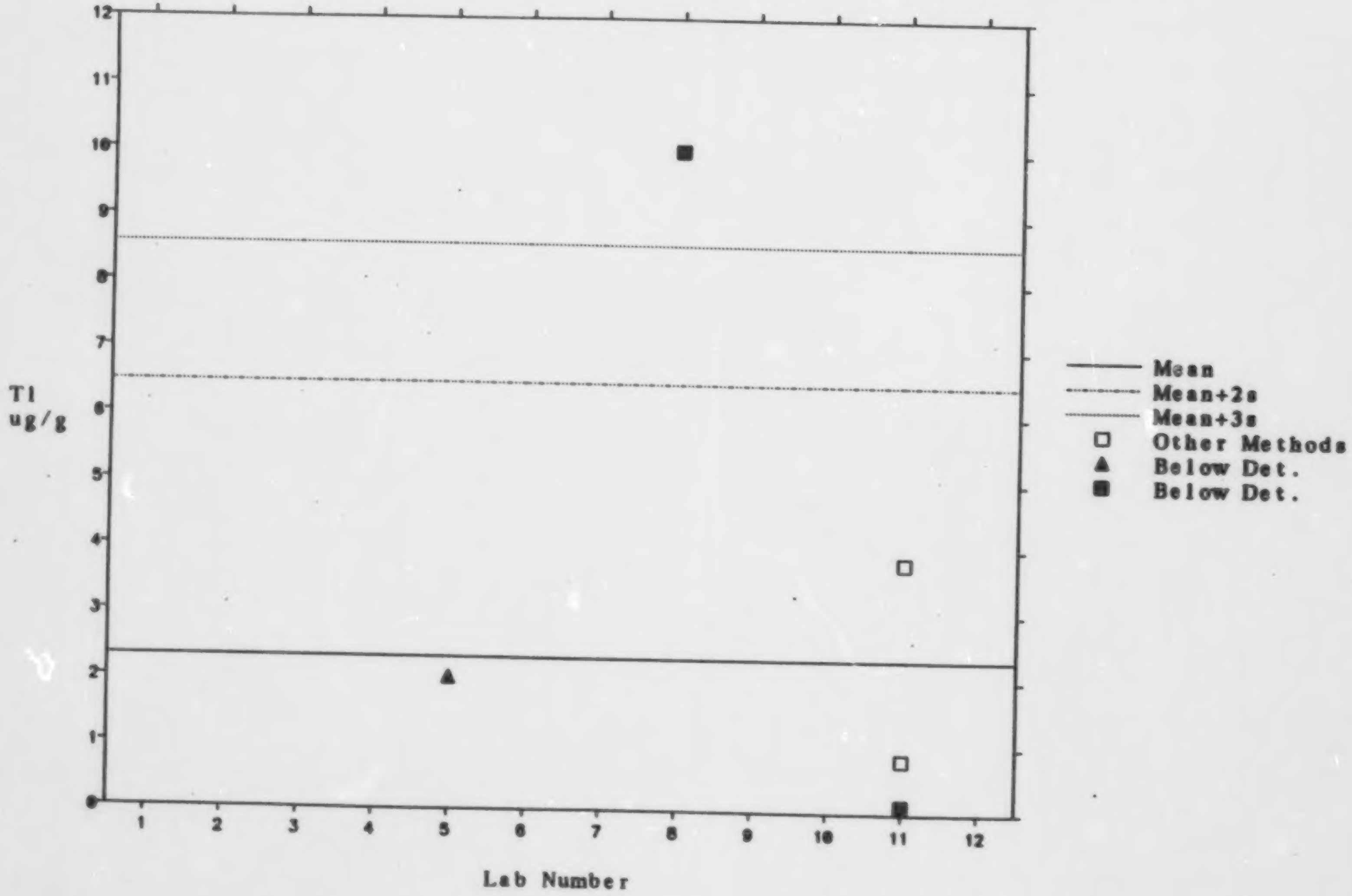


Figure 176. Analysis of Tl in Sludge F

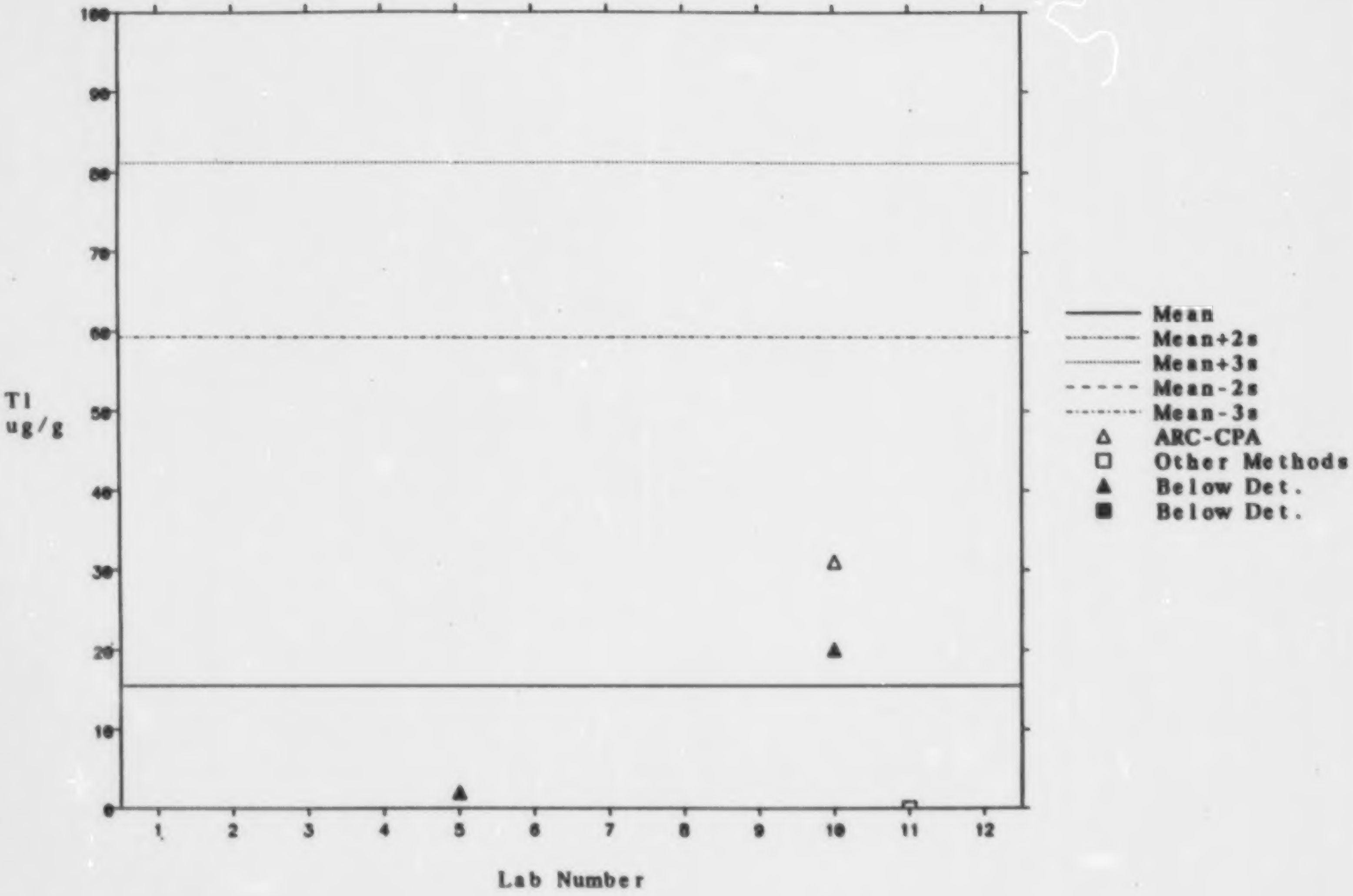
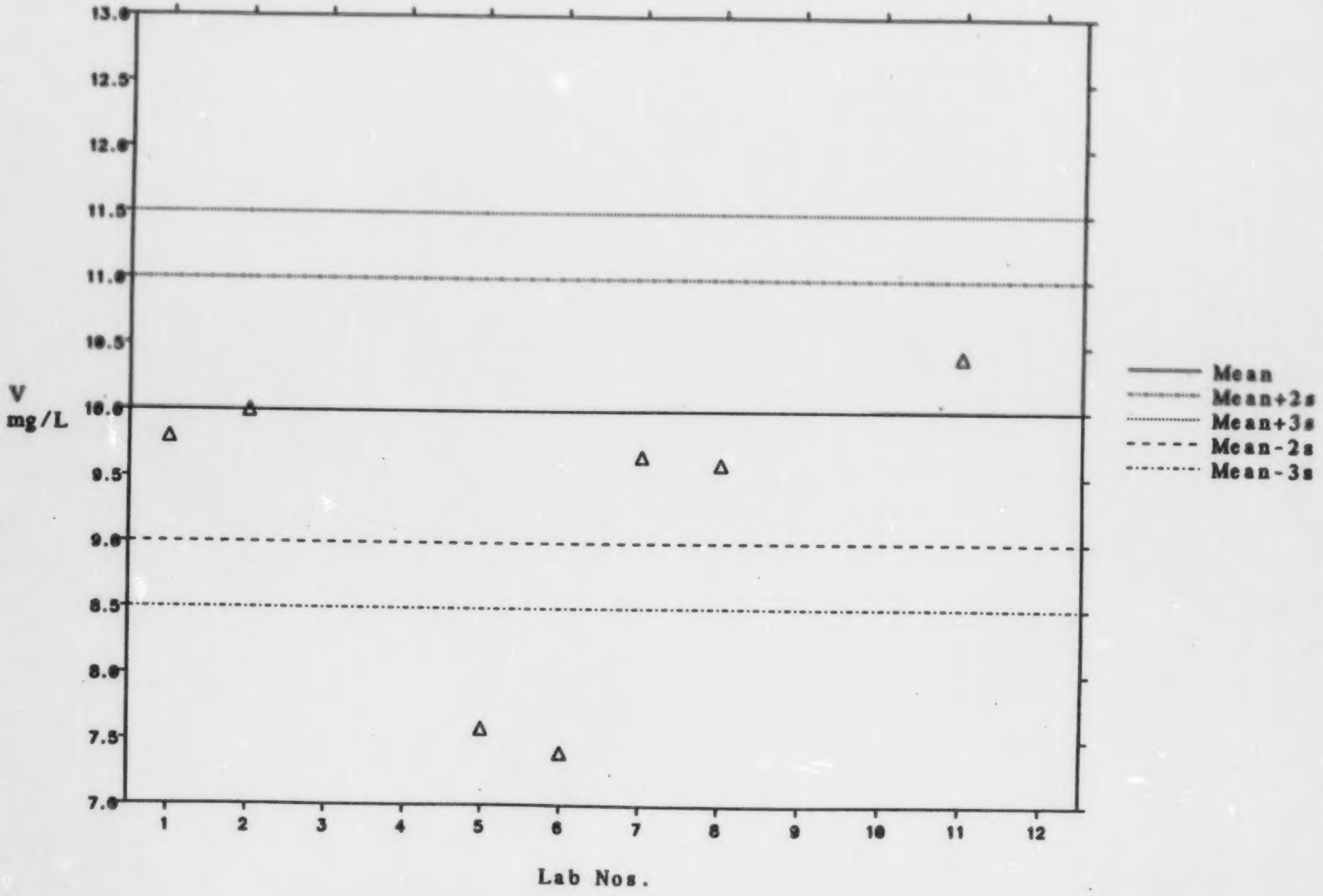


Figure 177. Mean and Confidence Intervals for V in an ICP Standard



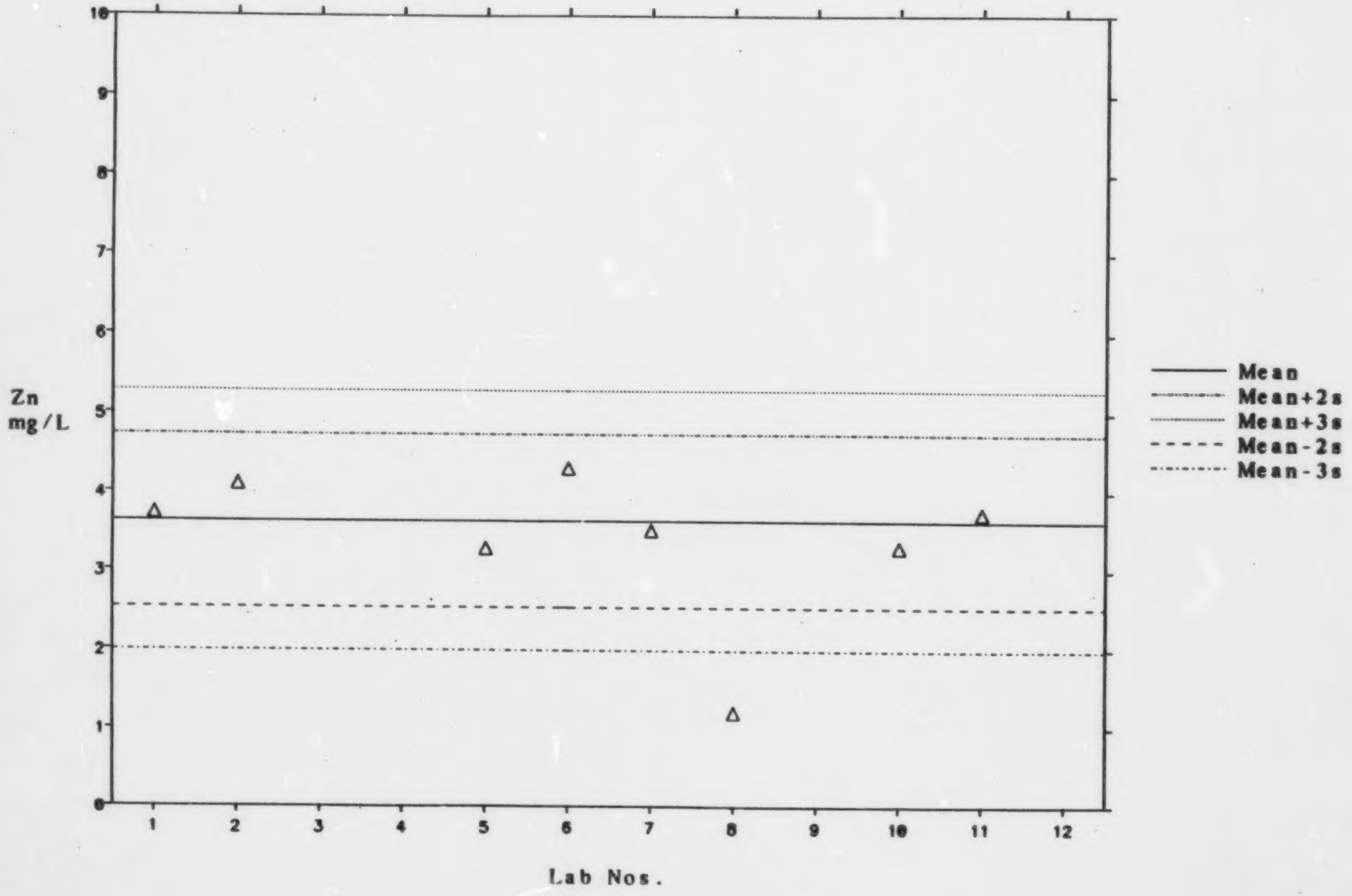
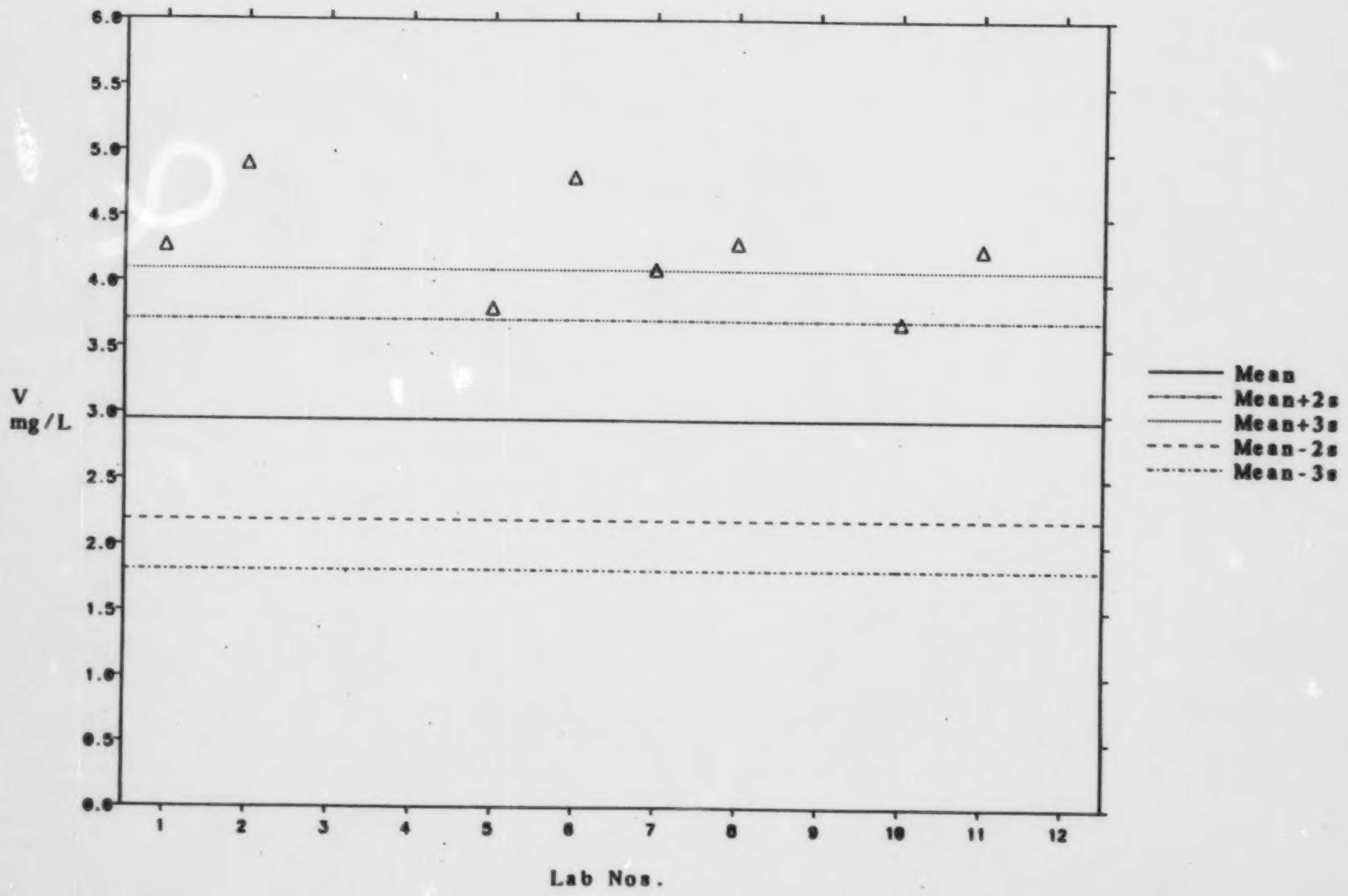


Figure 178. Mean and Confidence Intervals for V in Digestate 3

Figure 179. Mean and Confidence Intervals for V in Digestate 4



within the warning limits, and 1 lab outside the control limits. Five labs were outside the control limits of Digestate 4 (Figure 179), and only 3 labs were within these limits. A probable explanation of the discrepancy between the digestate A and B results could be that the V concentration in digestate 4 is higher than calculated, as the outlier labs in digestate 4 were all close in values and higher than the control limit. Seven labs reported V for the standard A and eight labs for the standard B samples (Figures 180 and 181). Figure 180 shows that 6 labs reported data within Standards A's control limits and the other one was lower than the limit. Standard B values (Figure 181) show that all but two labs had data within the control limits, with the 2 labs reporting data higher than the control limit. Figures 180 and 181 also show that the results of the ARC-CPA method were not different than those reported by other methods.

The statistical data for V in sludges A to F are shown in Table 19, and their control charts in Figures 183 to 187. Table 19 shows that results (above detection, were reported for all sludges, with number of analyses varying from 12 to 36 samples. The mean V values ranged from 4.7 mg/kg for sludge E to 62 for sludge B. The coefficient of variation of the sludges varied from 25 to 60% and it seems not to be related to either the number of reported results or the measured concentrations.

Figures 183 to 187 indicate that all the reported V data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The V data shows good recovery of the element for some of the standards, and some agreement between the different labs and for the different methodologies used. The variability of the sludge values did not seems to be dependent on the number of analyses performed or the V concentration as found with some other elements.

4.1.1.19 Zn

Seven labs reported Zn in the ICP standard (Figure 188), all but two labs were within the control limits. The two outlier labs were both lower than the control limit. Eight labs reported Zn for the digestate

Figure 180. Mean and Confidence Intervals for V in Standard A

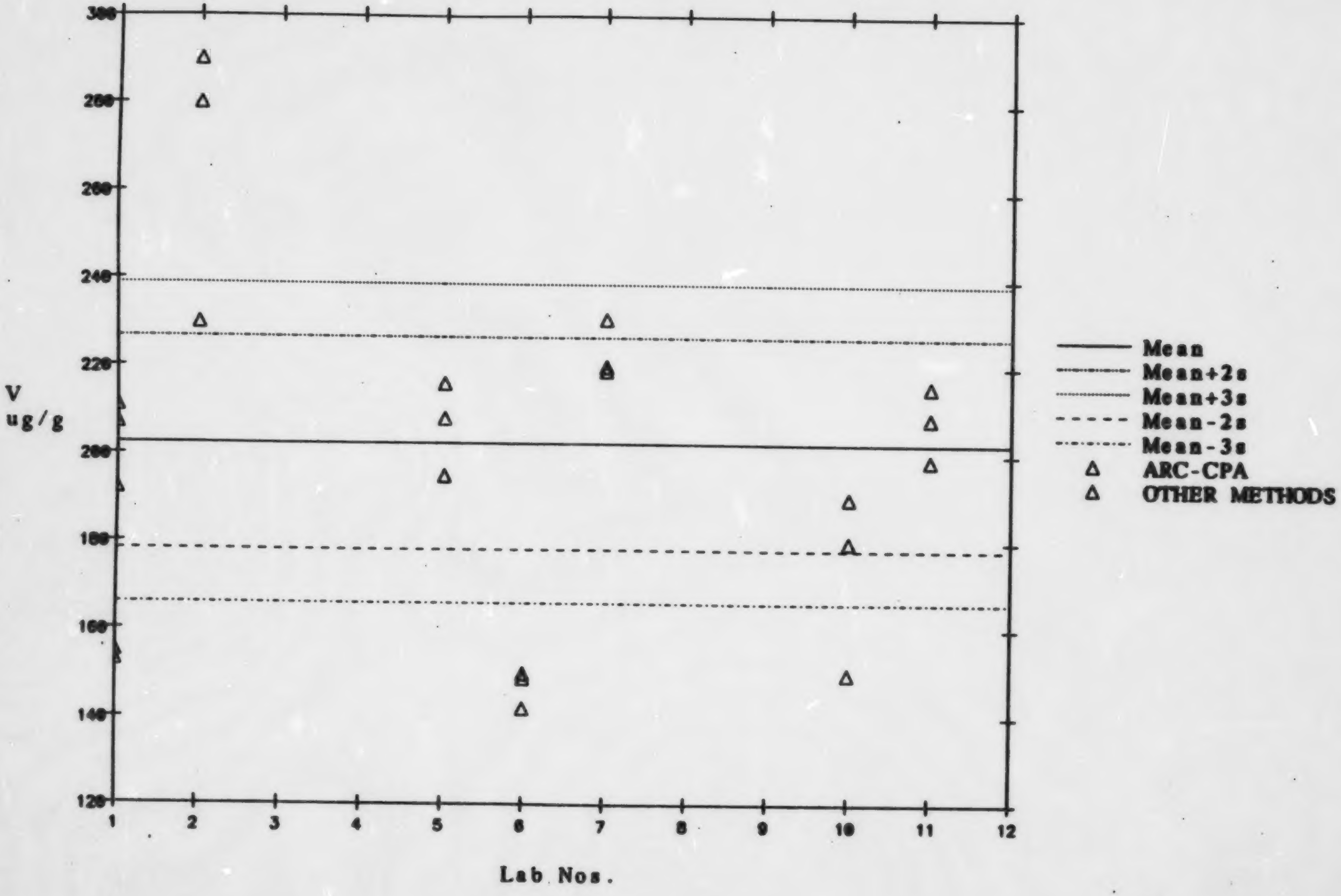


Figure 181. Mean and Confidence Intervals for V in Standard B

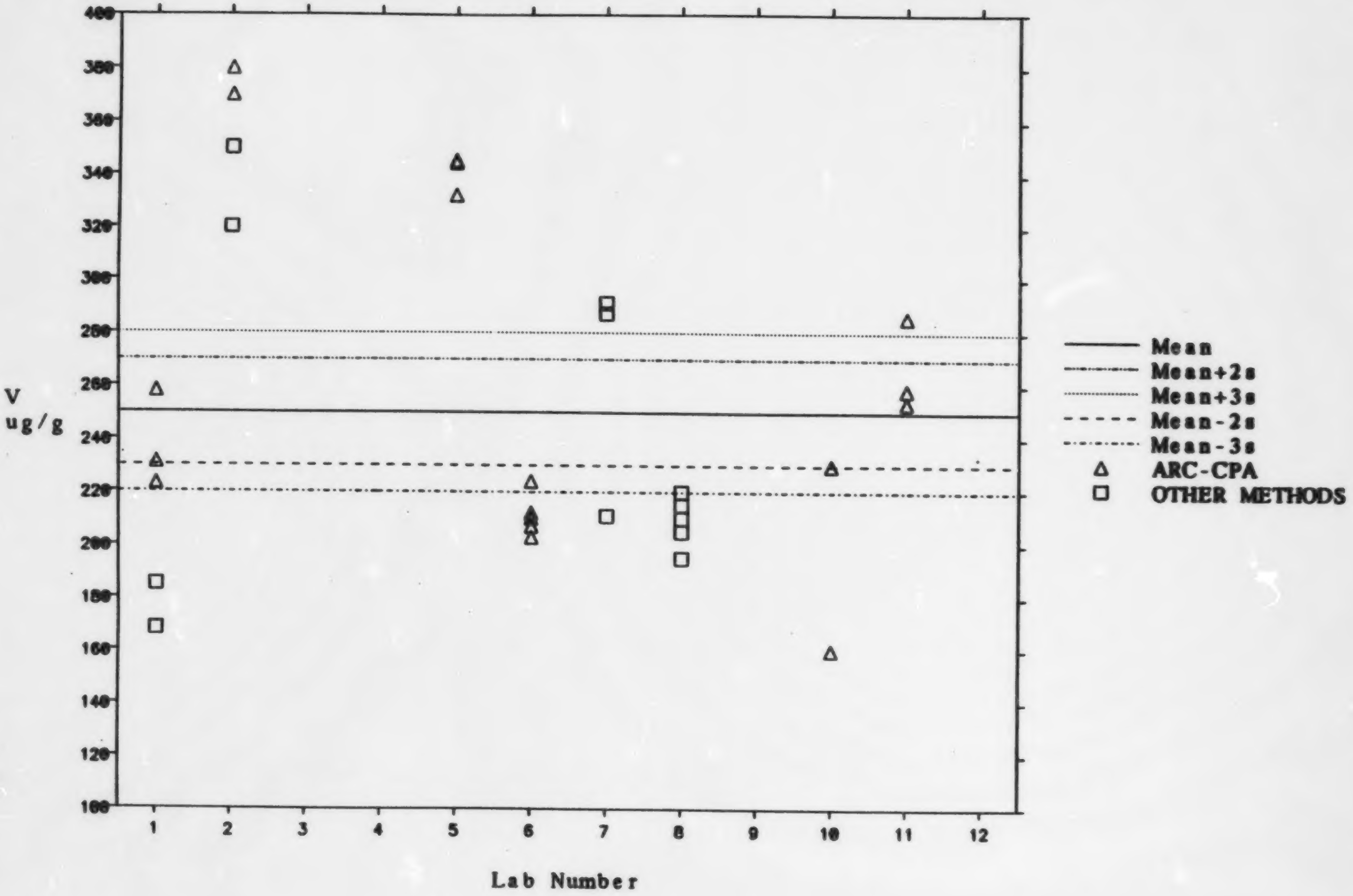


Figure 182. **Analysis of V in Sludge A**

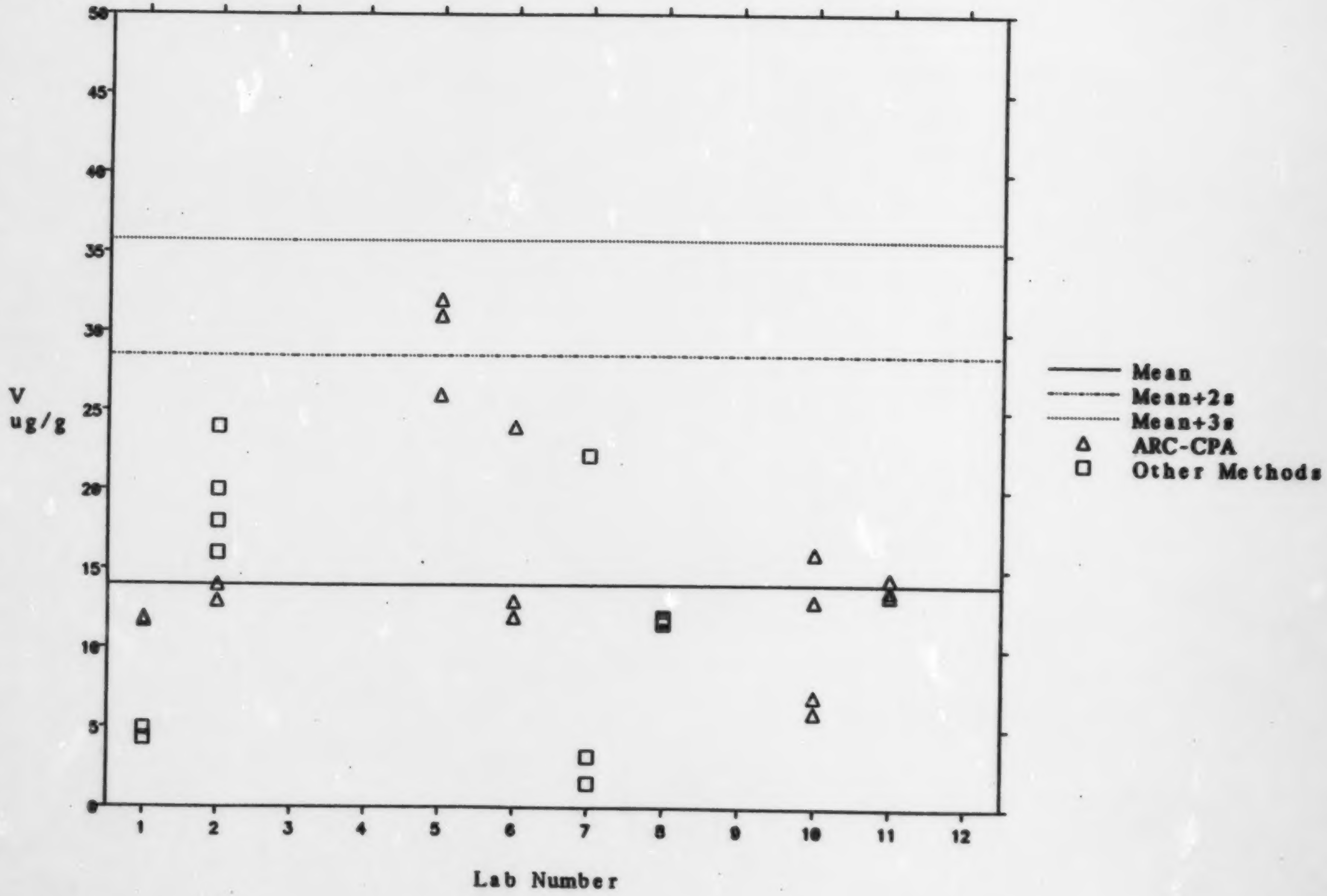


Figure 183. Analysis of V in Sludge B

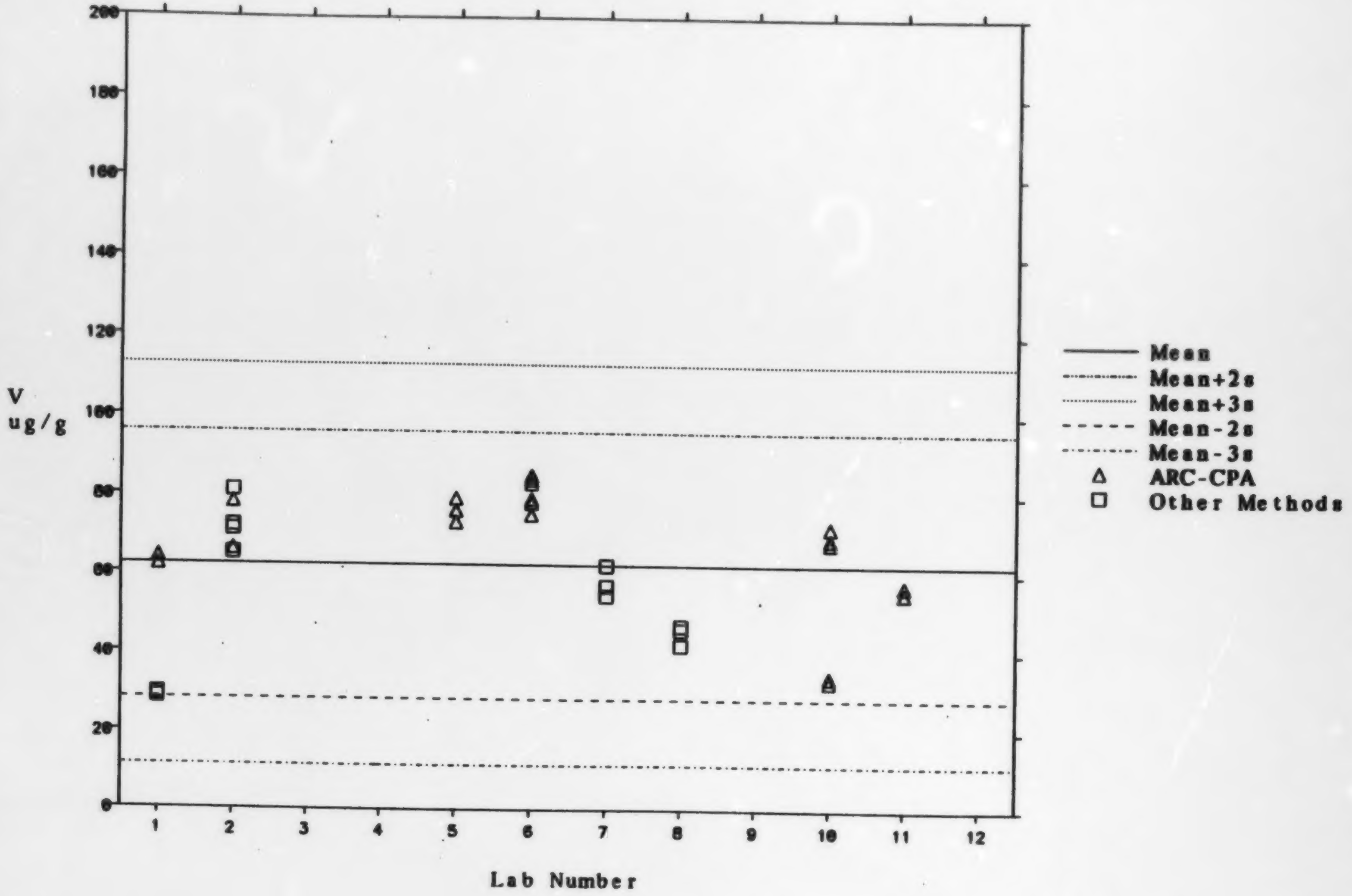


Figure 184. Analysis of V in Sludge C

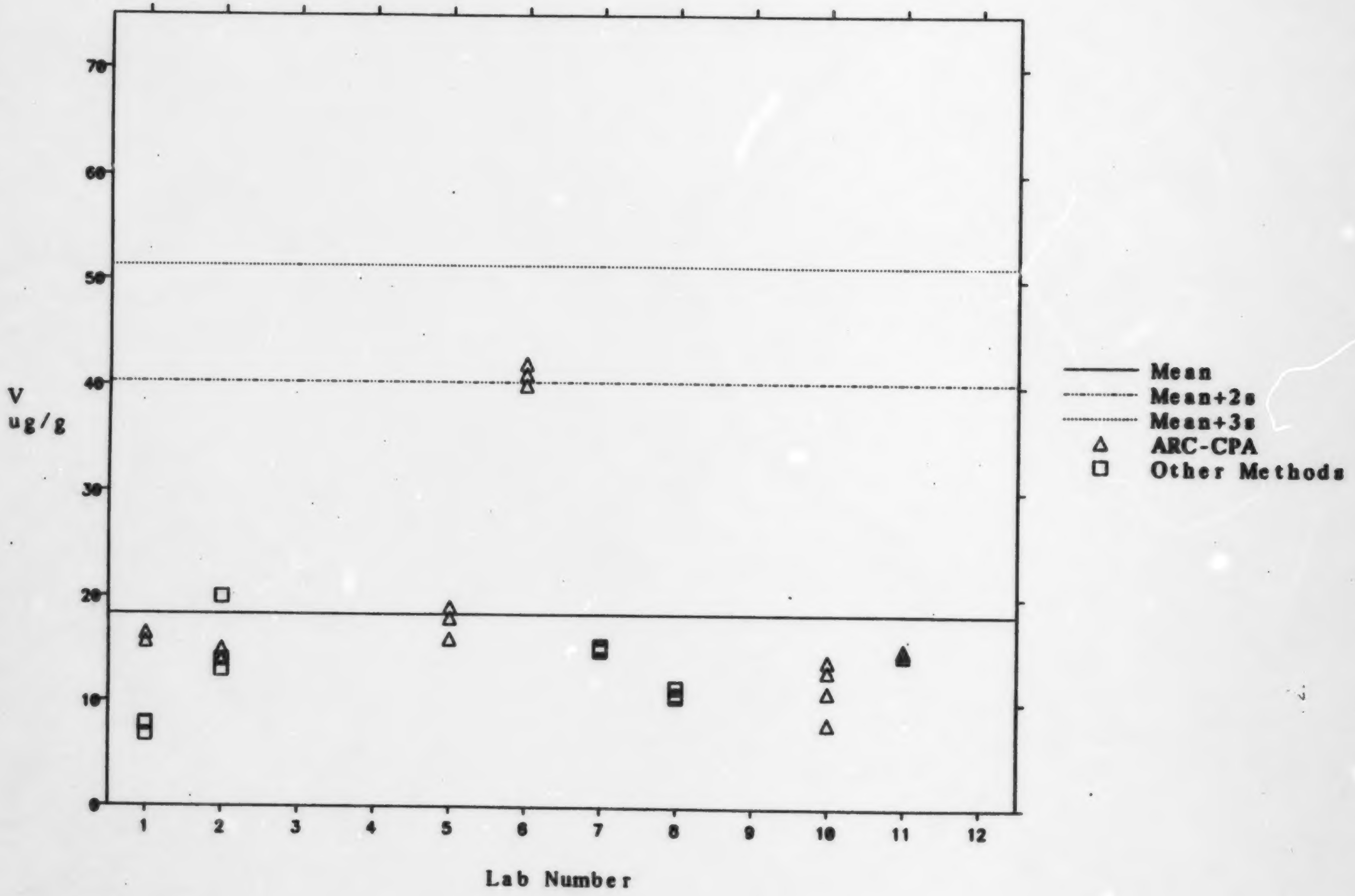


Figure 185. Analysis of V in Sludge D

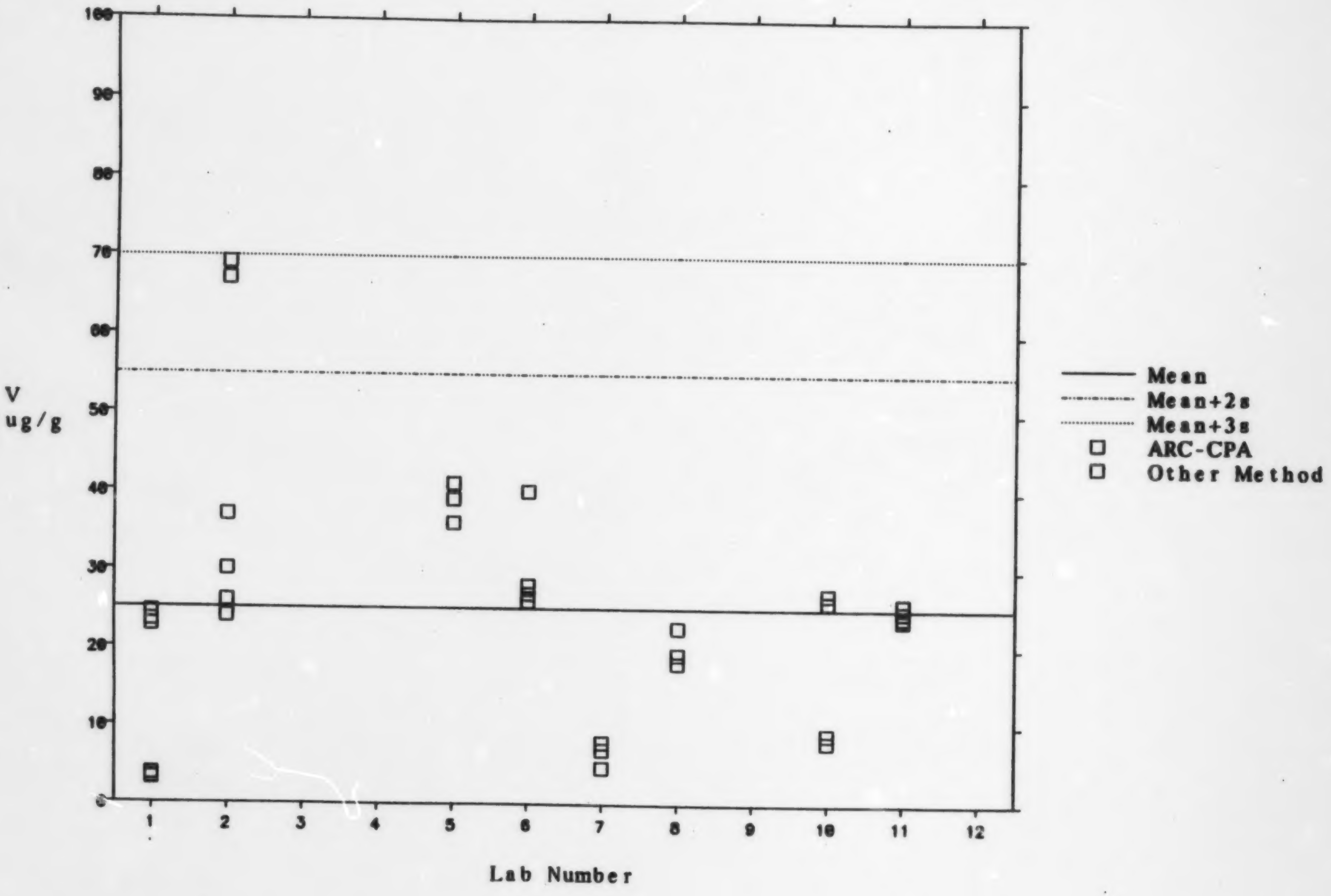


Figure 186. Analysis of V in Sludge E

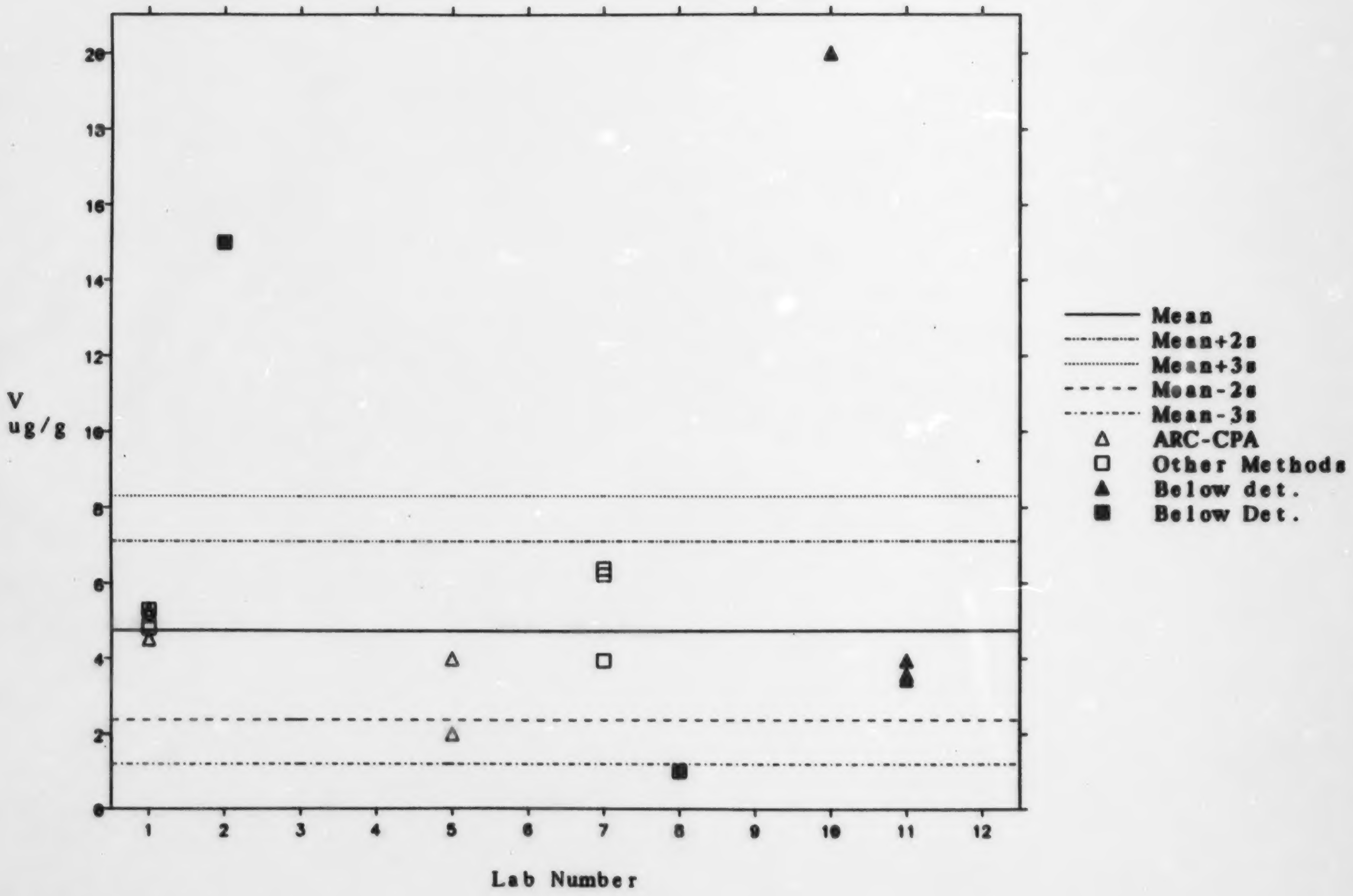


Figure 187. Analysis of V in Sludge F

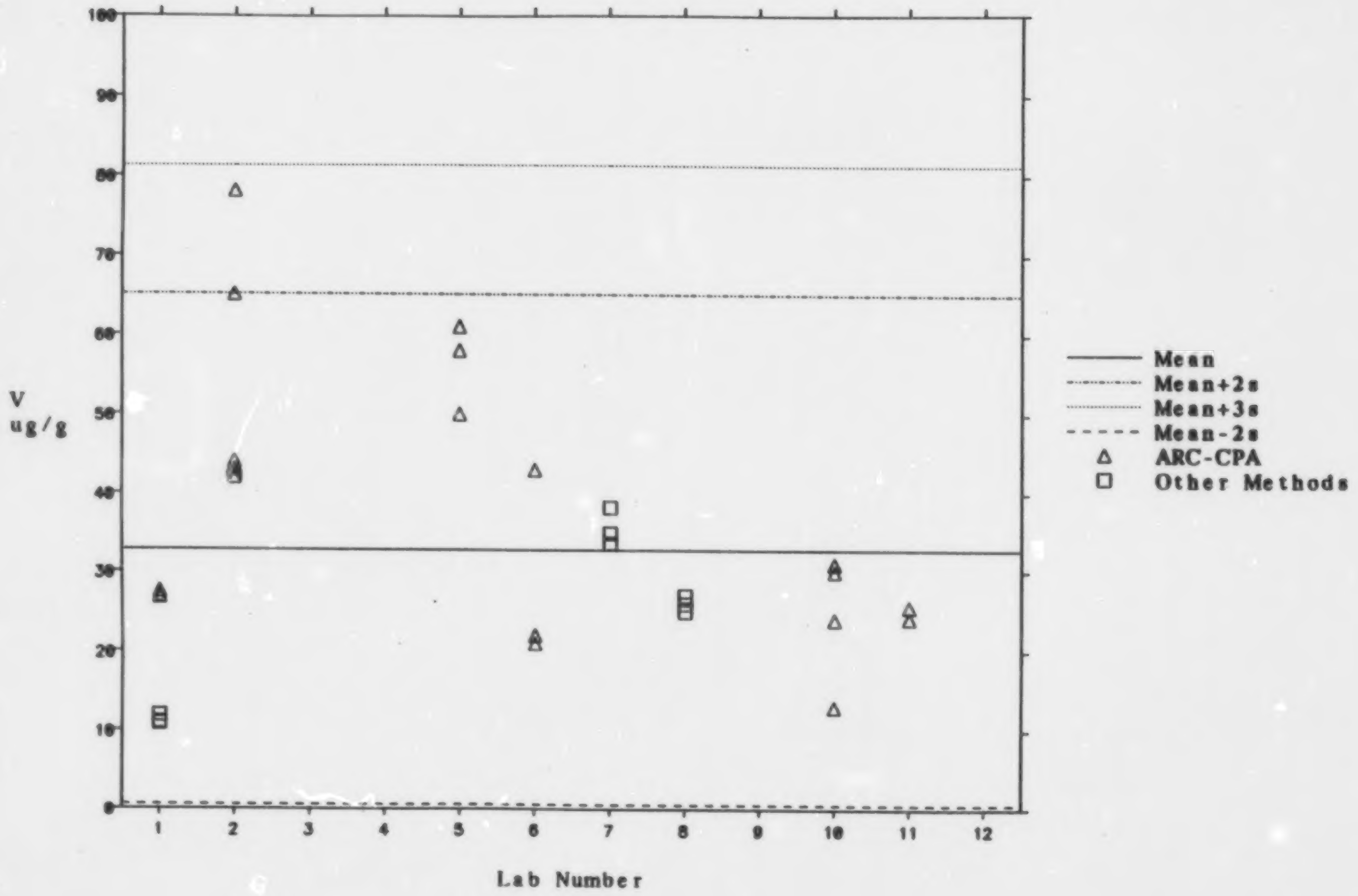
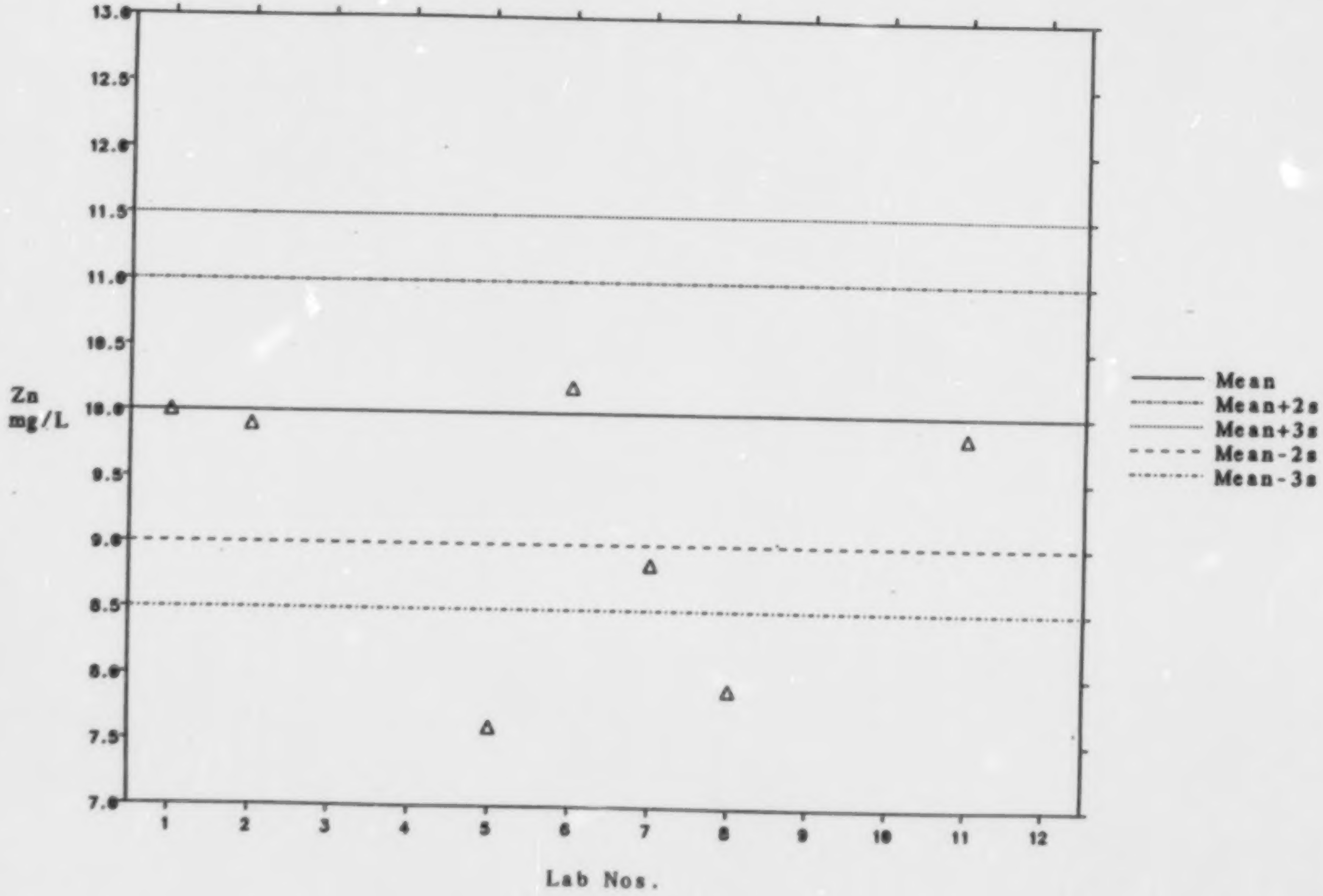


Table 19. Statistical Parameters for V (mg/Kg) in Sludges A-F.

Statistic	Sludge					
	A	B	C	D	E	F
Count	35.00	35.0	35.00	36.00	12.000	34.0
Sum	490.00	2170.0	642.00	902.00	57.000	1120.0
Mean	14.00	62.1	18.30	25.00	4.750	32.8
SEM	1.22	2.9	1.85	2.49	0.342	2.8
Median	13.00	65.0	15.00	25.20	5.000	27.3
Variance	52.40	287.0	120.00	222.00	1.400	260.0
StDev	7.24	16.9	11.00	14.90	1.180	16.1
Maximum	32.00	85.0	42.00	69.00	6.390	78.0
Minimum	1.51	28.5	6.80	3.20	2.000	10.8
Range	30.50	56.5	35.20	65.80	4.390	67.2
Skewness	0.72	-0.7	1.45	1.03	-0.907	0.9
Kurtosis	0.58	-0.5	0.77	2.34	1.640	0.7
CV(%)	51.70	27.3	59.80	59.60	24.900	49.2

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 188. Mean and Confidence Intervals for Zn in an ICP Standard



samples (Figures 189 and 190). Digestate 3 and 4 data (Figures 189 and 190) were all within the warning limits, an indication of very good recovery of Zn and good agreement between all 8 labs. Eight labs reported Zn for the standard A and B samples (Figures 191 and 192). Figure 191 shows that all labs reported data within Standards A's warning limit and only 2 labs had data between the warning and control limits. Standard B values (Figure 192) show that five labs had data within the control limits, three labs were completely outside the control limits, and only two labs were completely within the control limits. The differences between Standard A and B results is likely due to the much smaller control limits of Standard B. Figures 191 and 192 also show that the results of the ARC-CPA method were not different from the results of other methods.

The statistical data for Zn in sludges A to F are shown in Table 20, and their control charts in Figures 189 to 198. Table 20 shows that results (above detection) were reported for all sludges, with number of analyses varying from 25 to 41 samples. The mean Zn values ranged from 7.5 mg/kg for sludge E to 2210 for sludge B. The coefficient of variation of the sludges varied from 15 to 77% and it seems to be inversely related to the number of reported results and the measured concentrations.

Figures 193 to 198 indicate that most of the reported Zn data fell within the sludges' control limits, with no differences between the ARC-CPA and other methods.

The Zn data shows good recovery for most of the standards, and some agreement between the different labs and for the different methods used. The variability seems to be dependent on the number of analyses performed and the Zn concentration, with lower CV attained when more samples are analyzed and/or higher concentrations are measured.

4.1.2 CYANIDE

The statistical data for CN in sludges A to F are shown in Table 21, and their control charts in Figures 199 to 204. Table 21 shows that results (above detection) were reported for all sludges, with number of analyses varying from 3 to 6 samples. The mean CN values ranged from

Figure 189: Mean and Confidence Intervals for Zn in Digestate 3

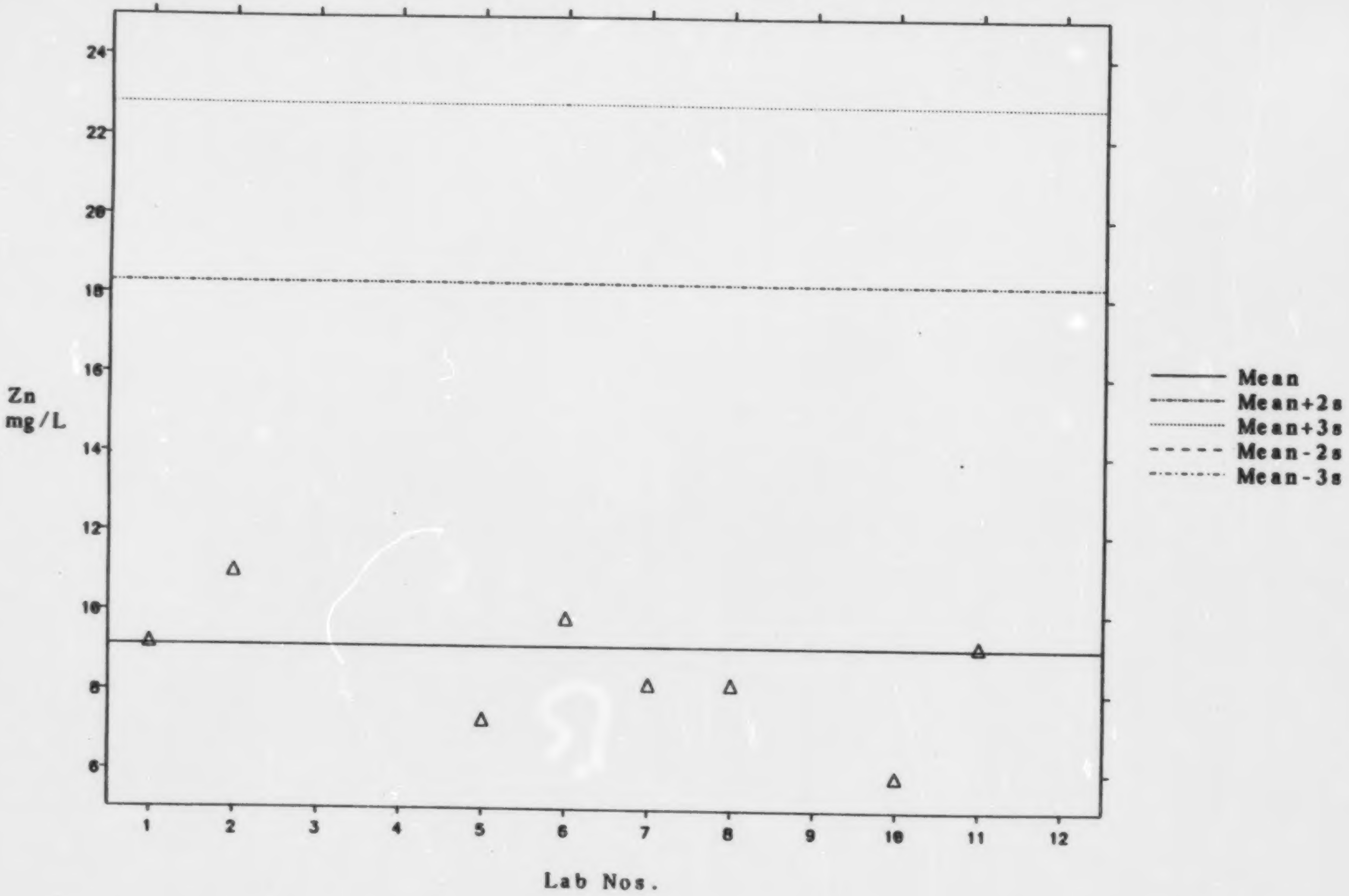


Figure 190. Mean and Confidence Intervals for Zn in Digestate 4

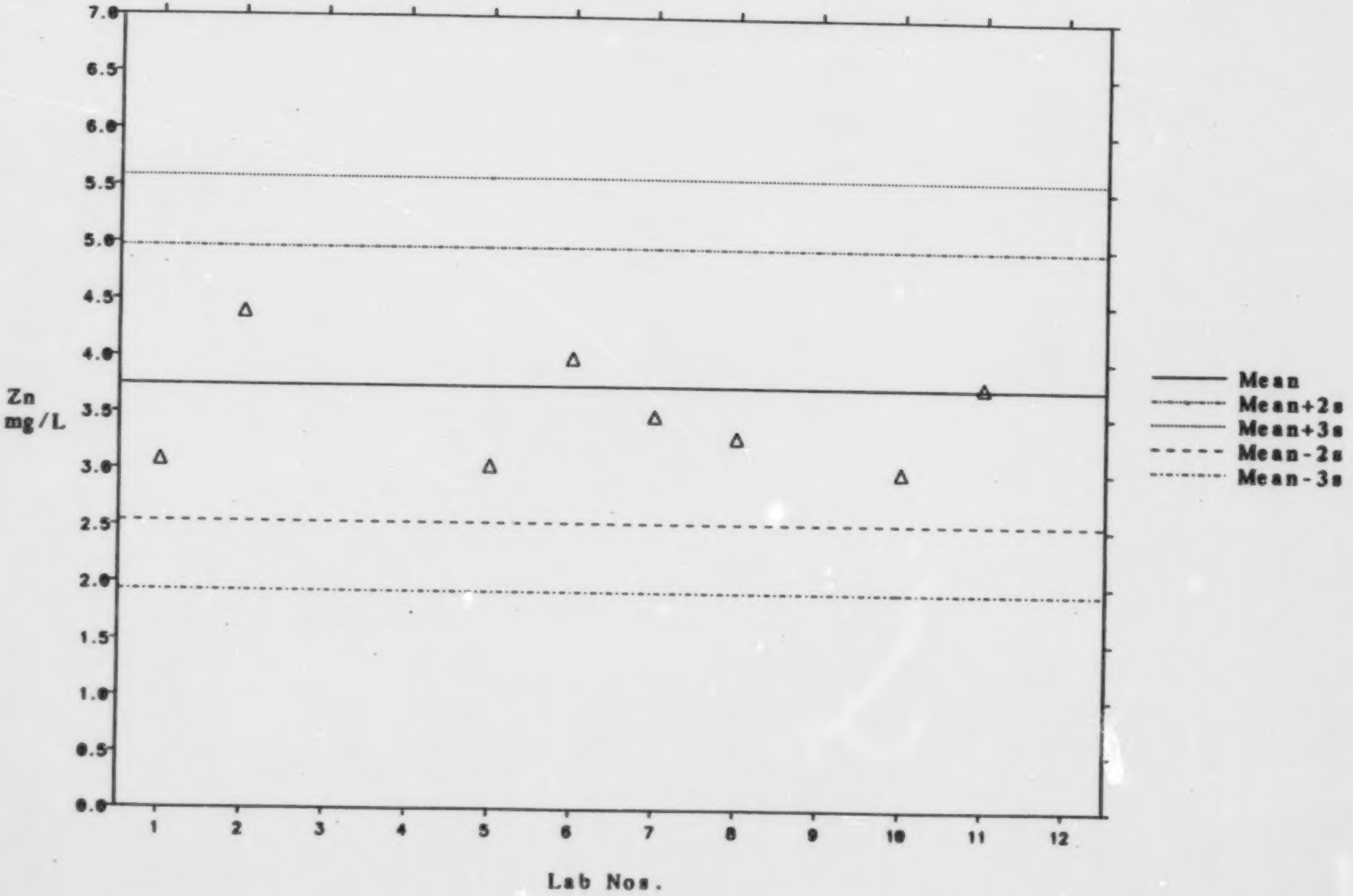


Figure 191. Mean and Confidence Intervals for Zn in Standard A

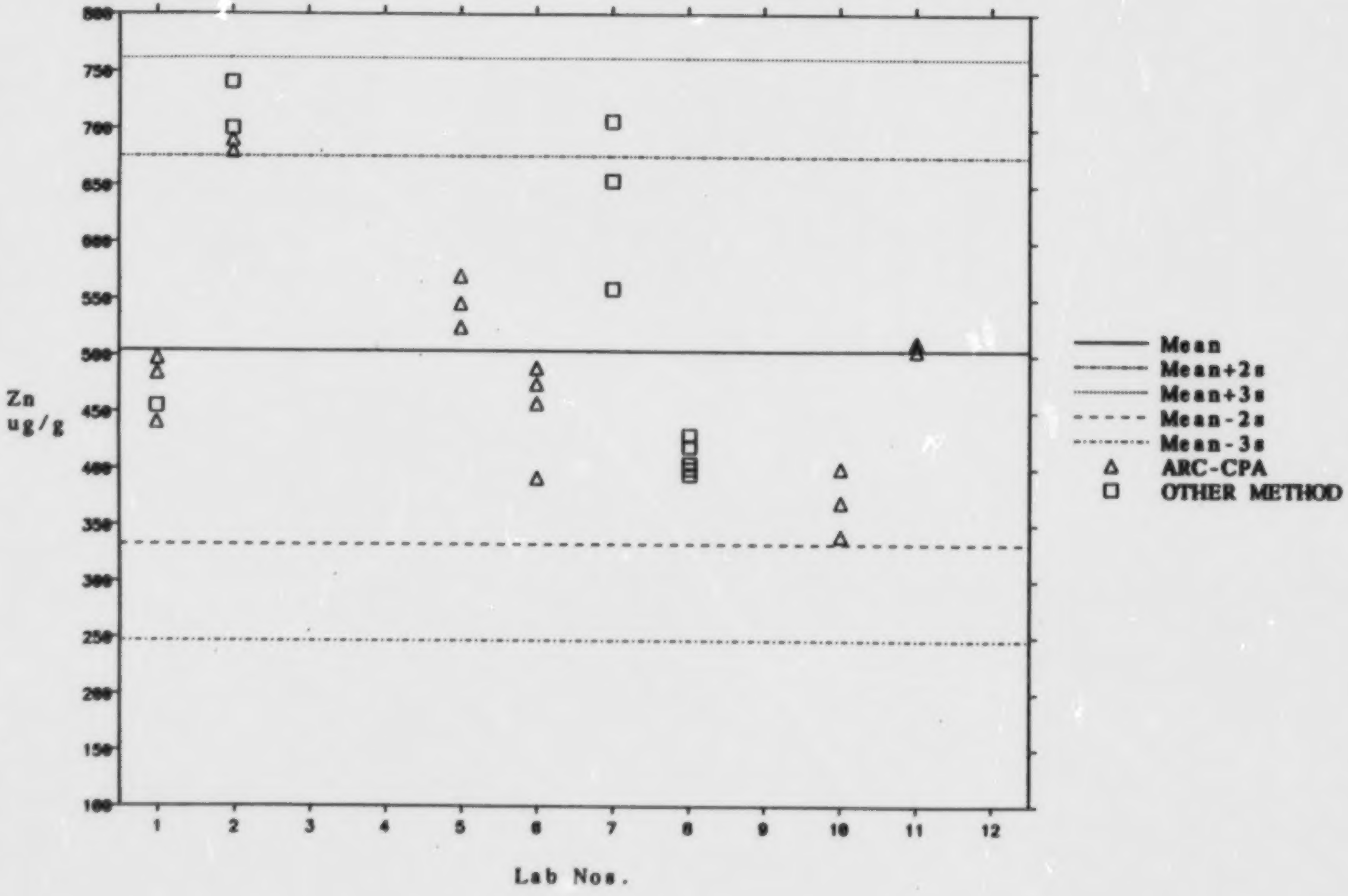


Figure 192. Mean and Confidence Intervals for Zn in Standard B

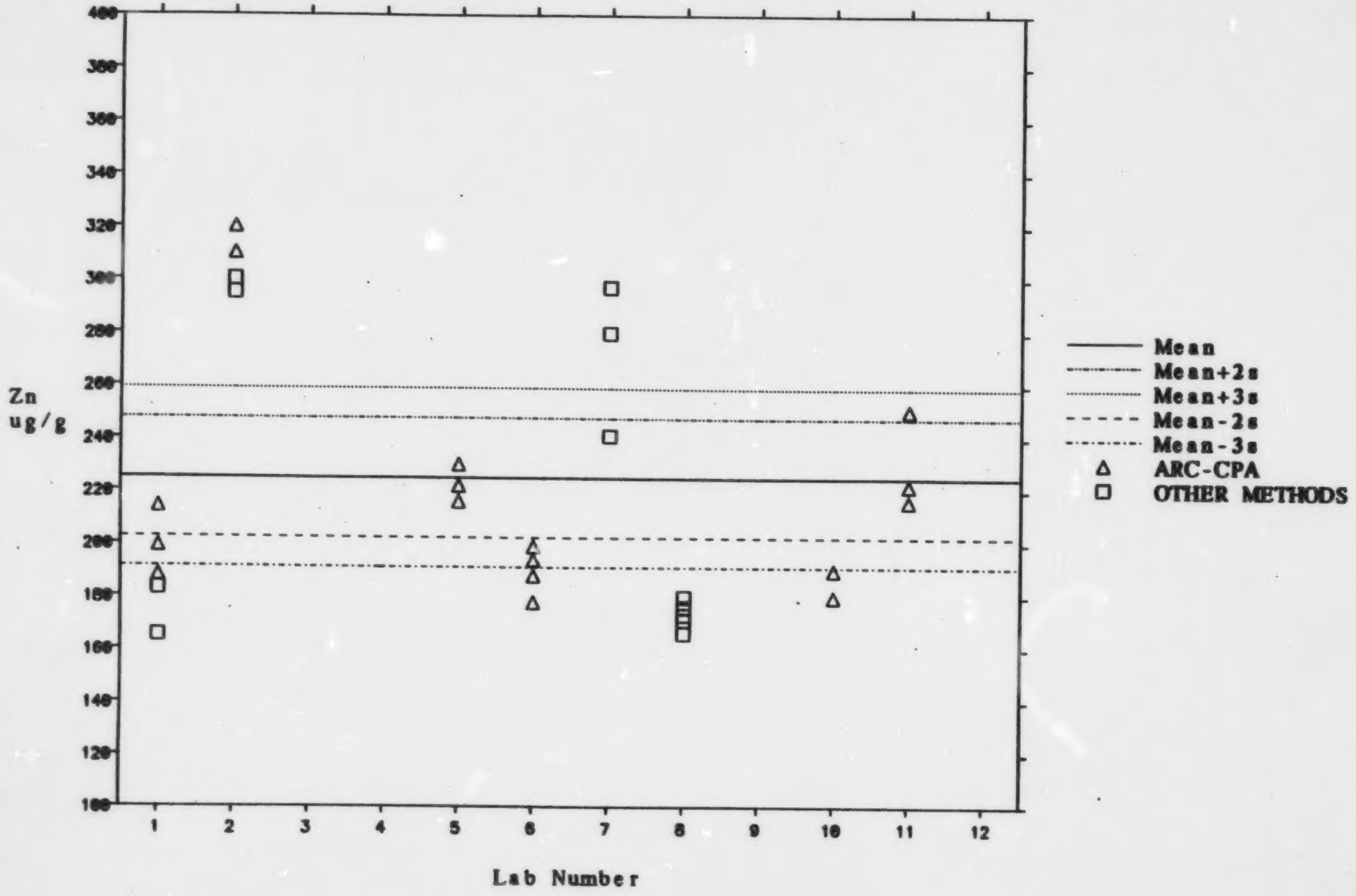


Table 20. Statistical Parameters for Zn (mg/Kg) in Sludges A-F.

Statistic	A	B	C	D	E	F
Count	36.0	41	35.0	42.0	25.00	34.0
Sum	1410.0	90500	3070.0	5290.0	187.00	4930.0
Mean	39.2	2210	87.7	126.0	7.46	145.0
SEM	2.6	51	2.7	6.6	1.15	7.7
Median	35.5	2220	86.0	110.0	4.40	120.0
Variance	247.0	106000	258.0	1850.0	33.30	2010.0
StDev	15.7	325	16.1	43.0	5.77	44.9
Maximum	85.5	3310	140.0	253.0	24.00	240.0
Minimum	15.1	888	69.0	66.0	3.30	94.0
Range	70.4	2420	71.0	187.0	20.70	146.0
Skewness	1.6	-9e-01	1.8	1.0	1.64	0.9
Kurtosis	2.8	9	3.8	0.7	2.01	-0.5
CV(%)	40.1	15	18.3	34.1	77.30	30.9

SEM=Standard error of mean; StDev= Standard deviation;
C.V=Coefficient of variability;

Figure 193. Analysis of Zn in Waste A

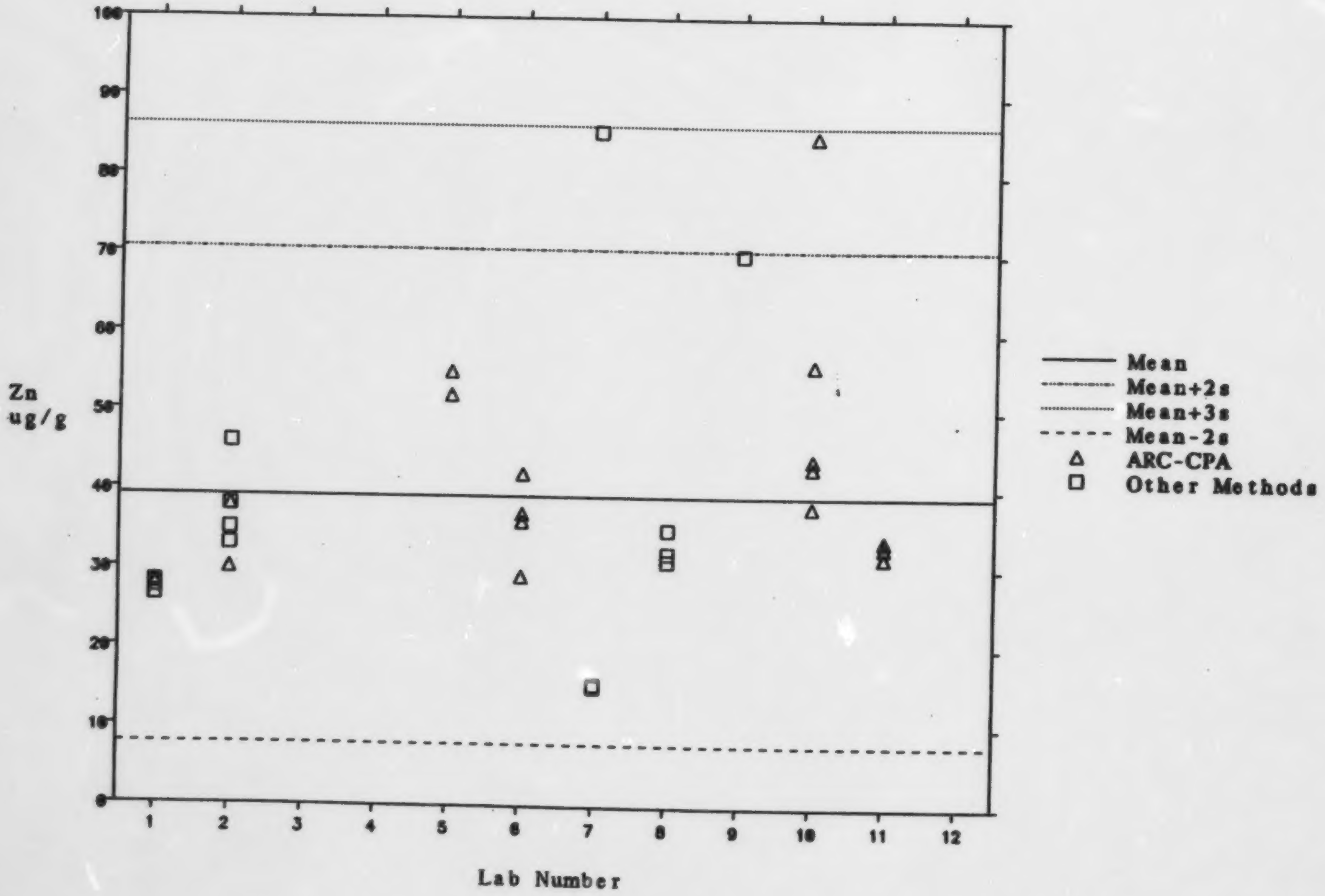


Figure 194. Analysis of Zn in Sludge B

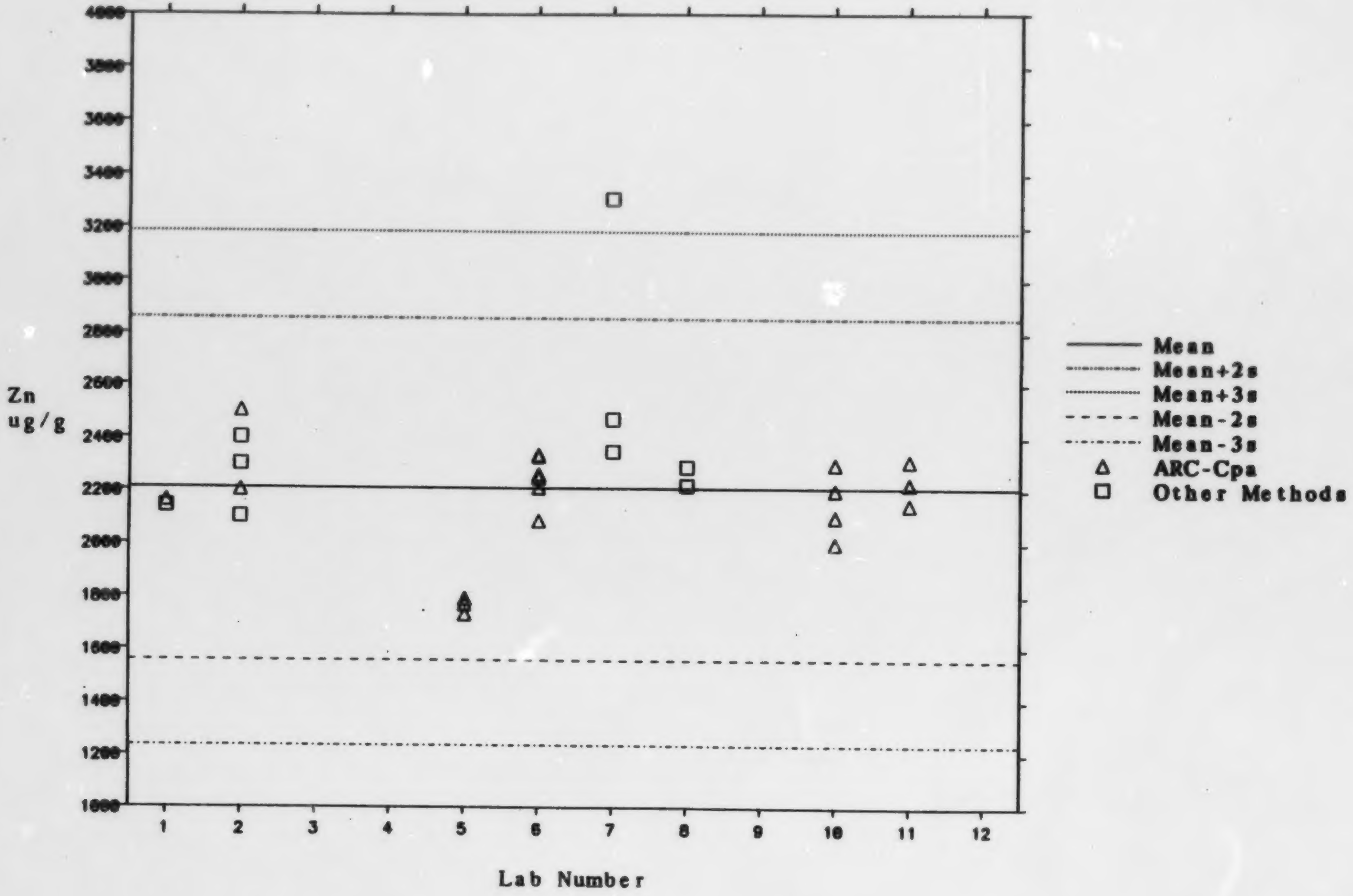


Figure 195. Analysis of Zn in Sludge C

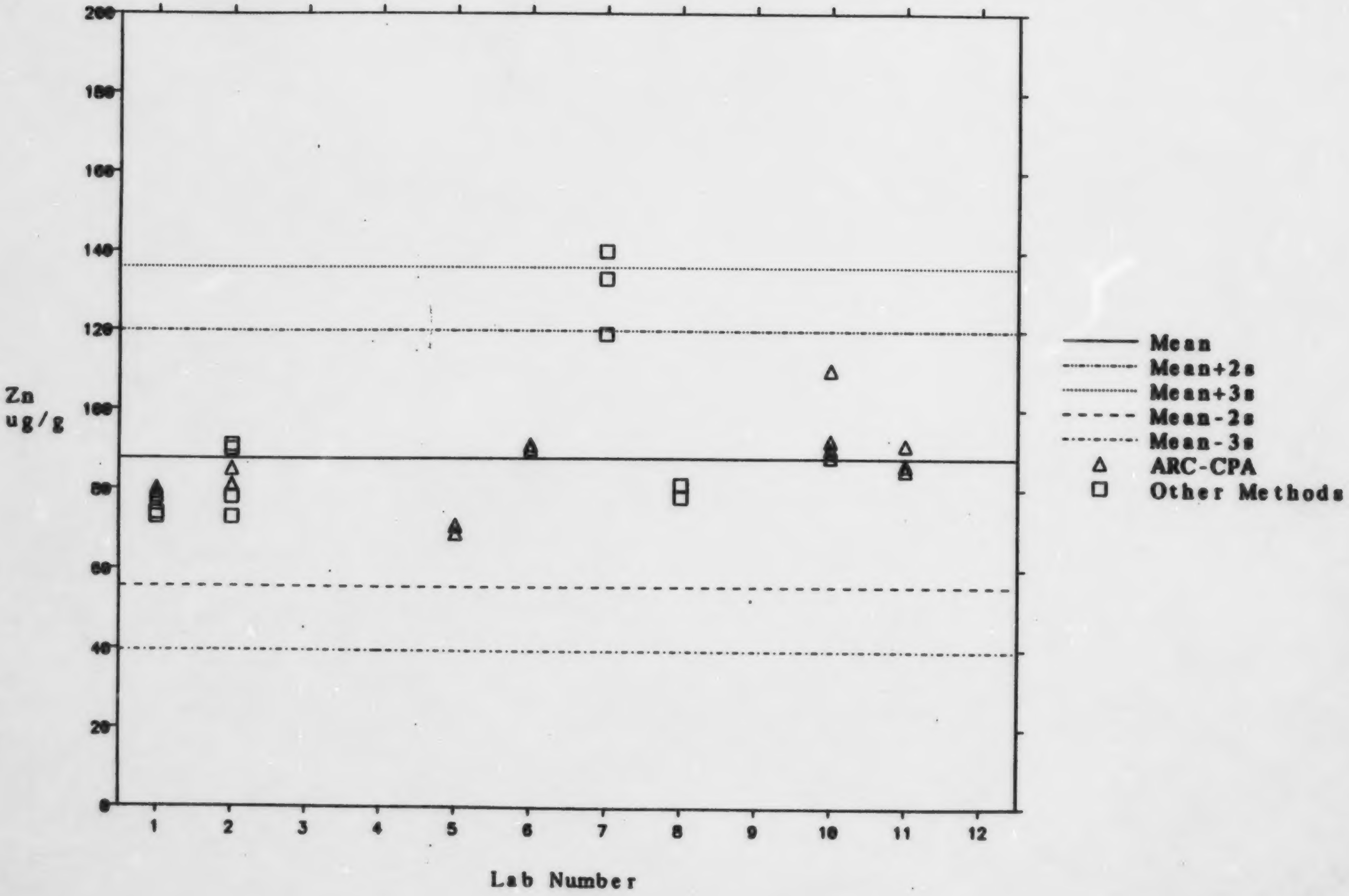


Figure 196. Analysis of Zn in Sludge D

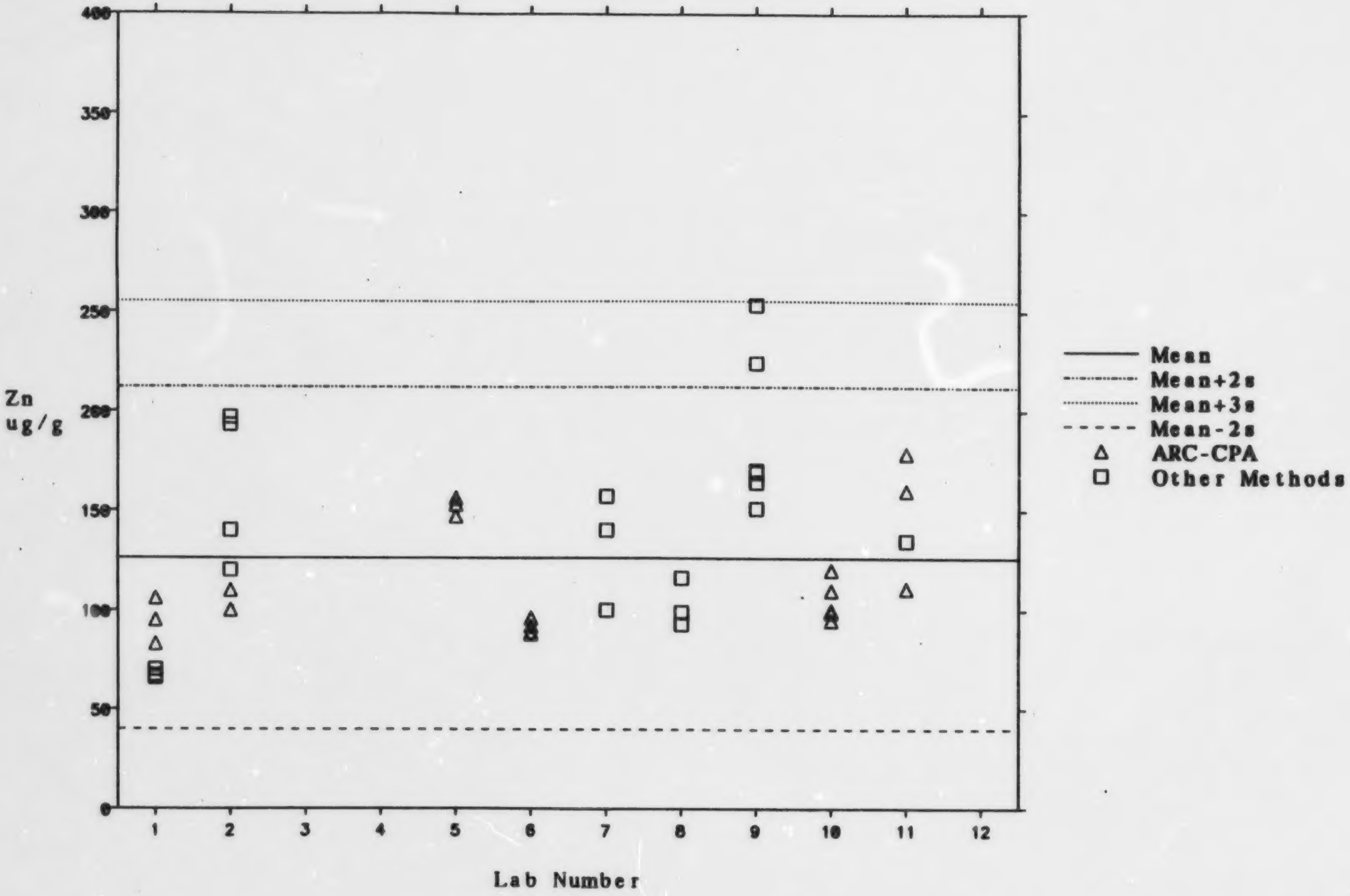


Figure 197. Analysis of Zn in Sludge E

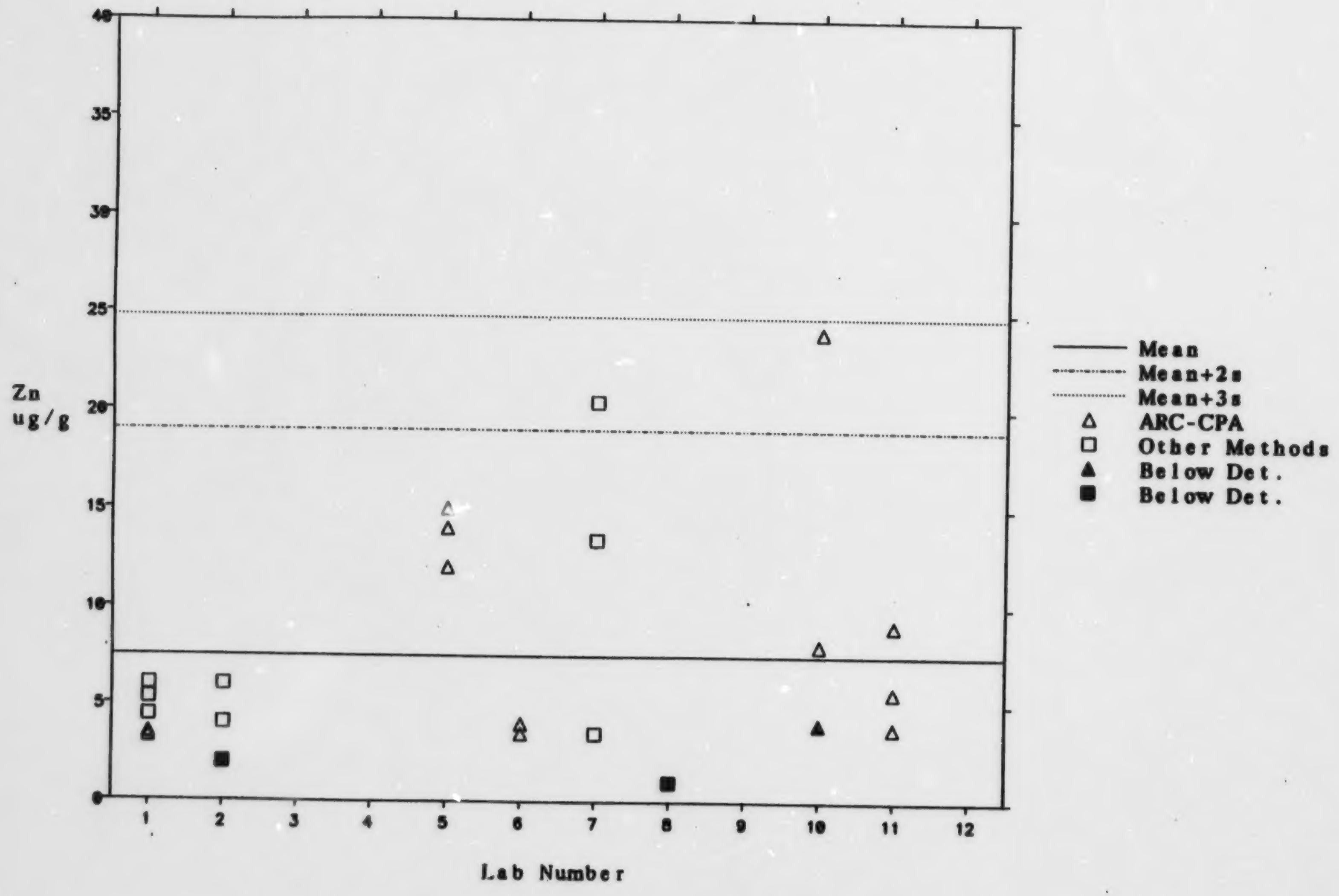


Figure 198. Analysis of Zn in Sludge F

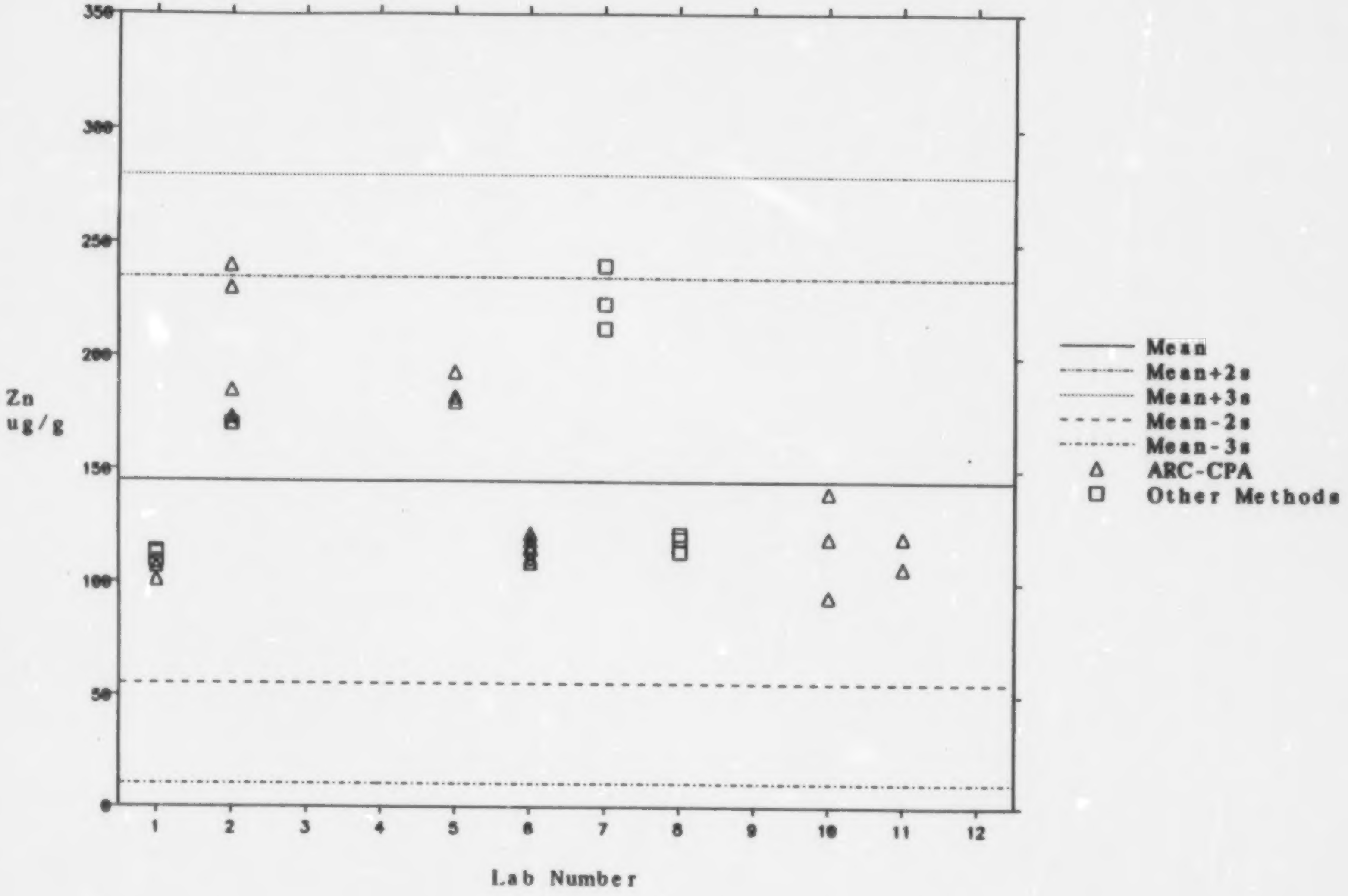


Table 21. Statistical Parameters for CN (mg/Kg) in Sludges A-F

Statistic	Sludge A	Sludge B	Sludge C	Sludge D	Sludge E	Sludge F
Count	4.00	5.00	6.00	4.00	4.00	3.00
Sum	173.33	33.82	44.45	1.87	14.79	3.90
Mean	43.33	6.76	7.41	0.47	3.70	1.30
SEM	42.56	5.39	6.37	0.18	3.11	0.49
Median	1.02	1.40	1.00	0.51	0.87	1.40
Variance	7244	145.10	243.28	0.13	38.61	0.73
StDev	85.11	12.05	15.60	0.35	6.21	0.85
Maximum	171.00	28.20	39.20	0.80	13.00	2.10
Minimum	0.30	0.22	0.25	0.05	0.06	0.40
Range	170.70	27.98	38.95	0.75	12.94	1.70
Skewness	2.00	2.18	2.43	-0.37	1.98	-0.52
Kurtosis	4.00	4.79	5.94	-3.47	3.93	
C.V(%)	196.42	178.09	210.54	75.80	168.04	65.72

SEM= Standard error of mean; StDev= Standard deviation;
C.V= Coefficient of variability;

Figure 199. Analysis of CN in Sludge A

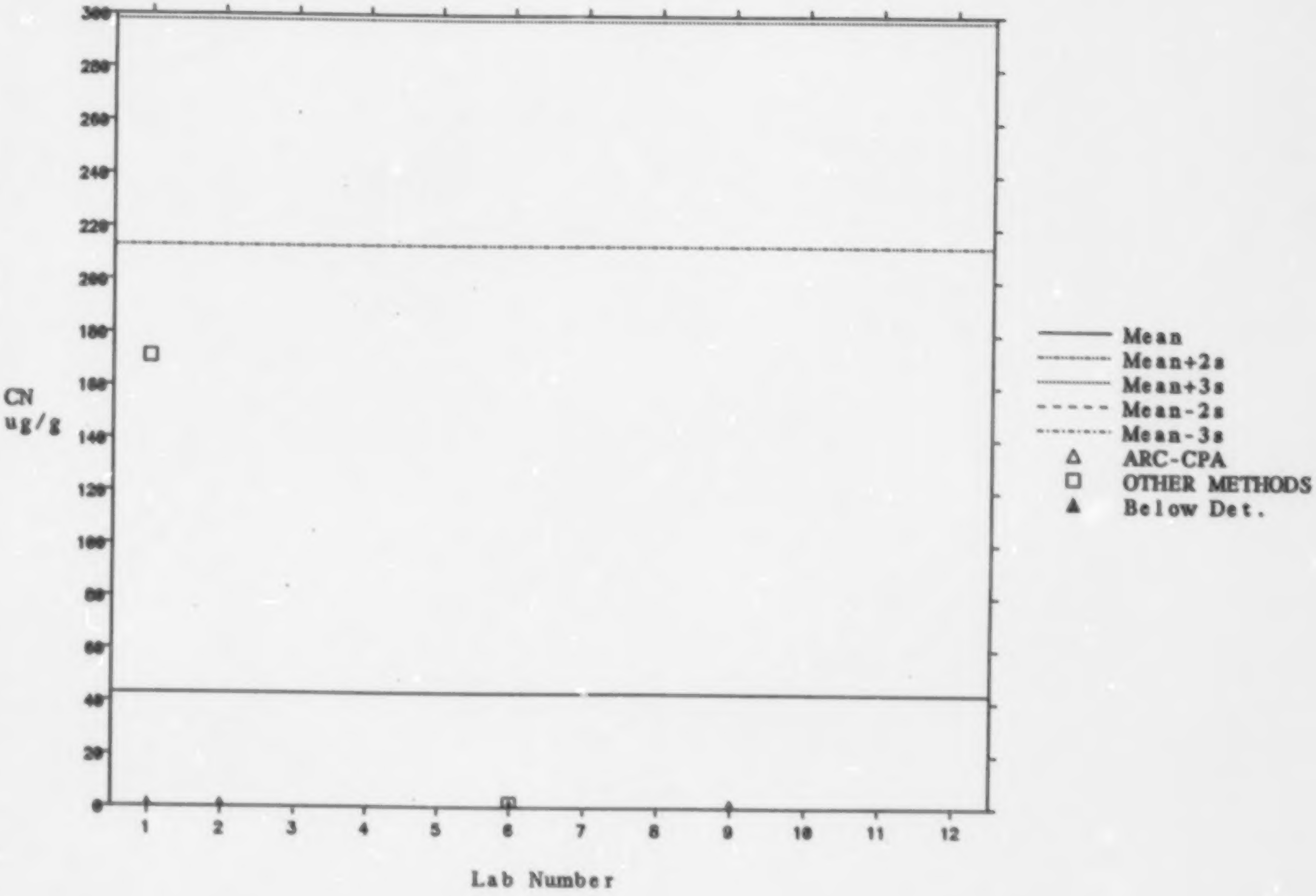


Figure 200. Analysis of CN in Sludge B

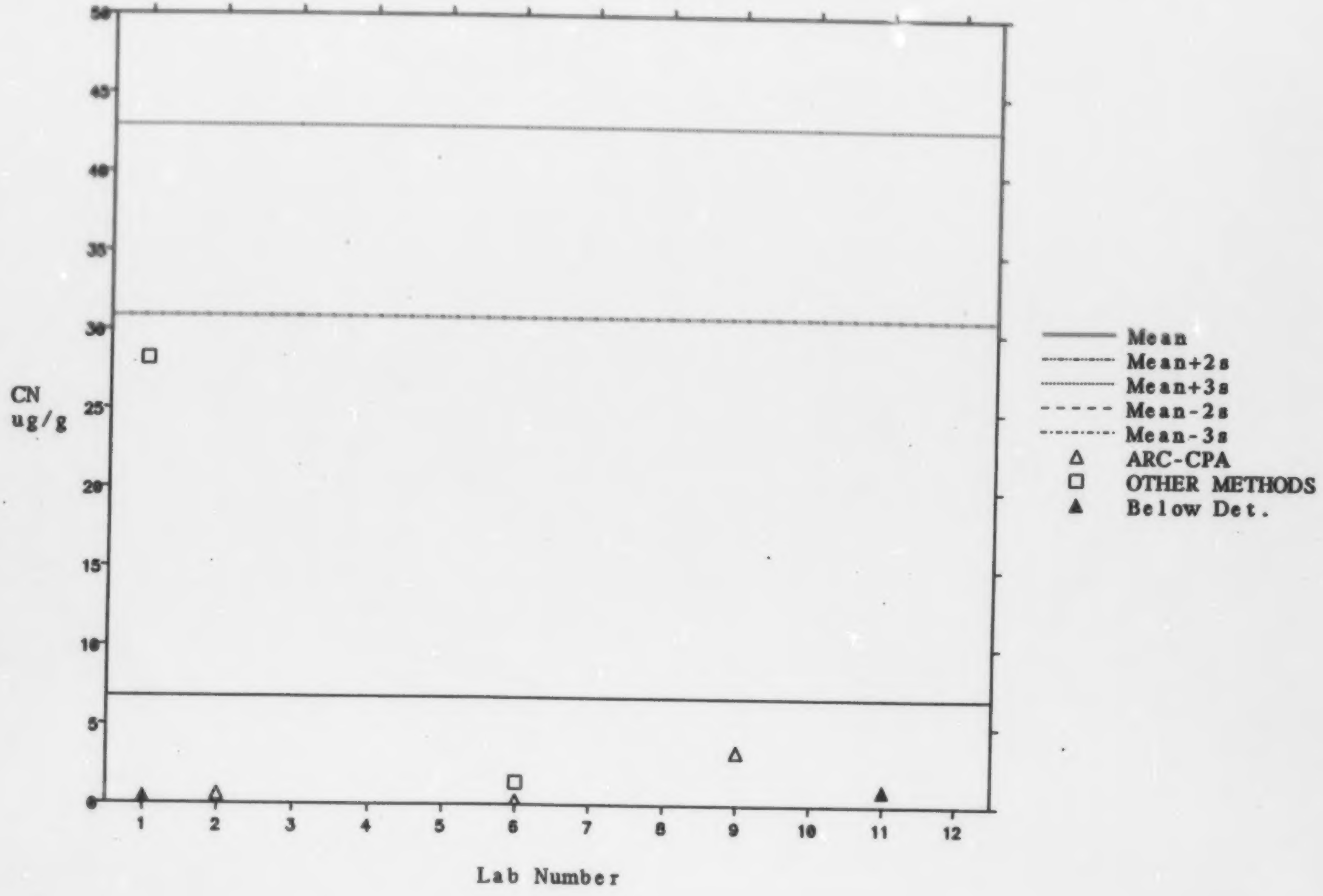


Figure 201. Analysis of CN in Sludge C

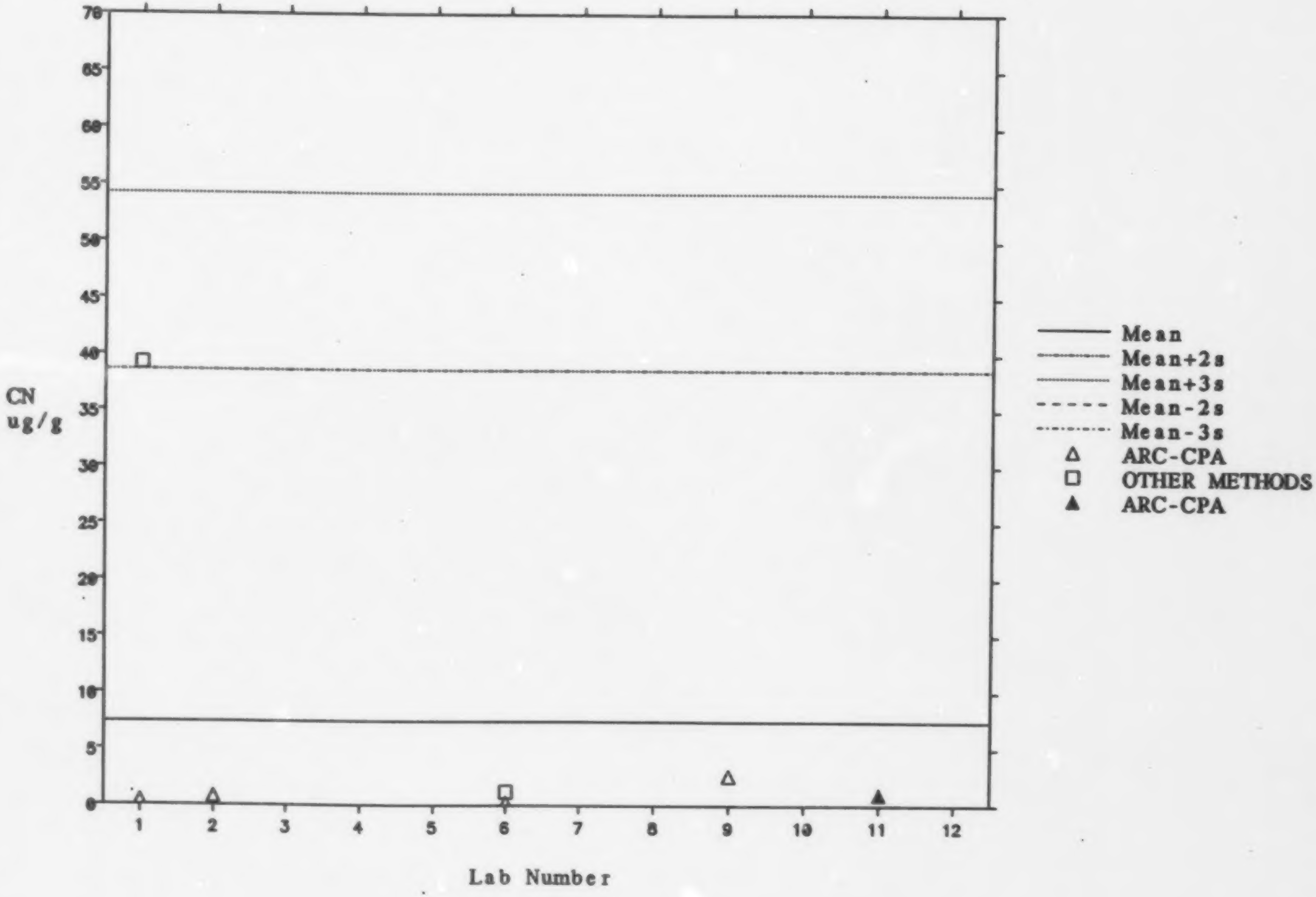


Figure 202. Analysis of CN in Sludge D

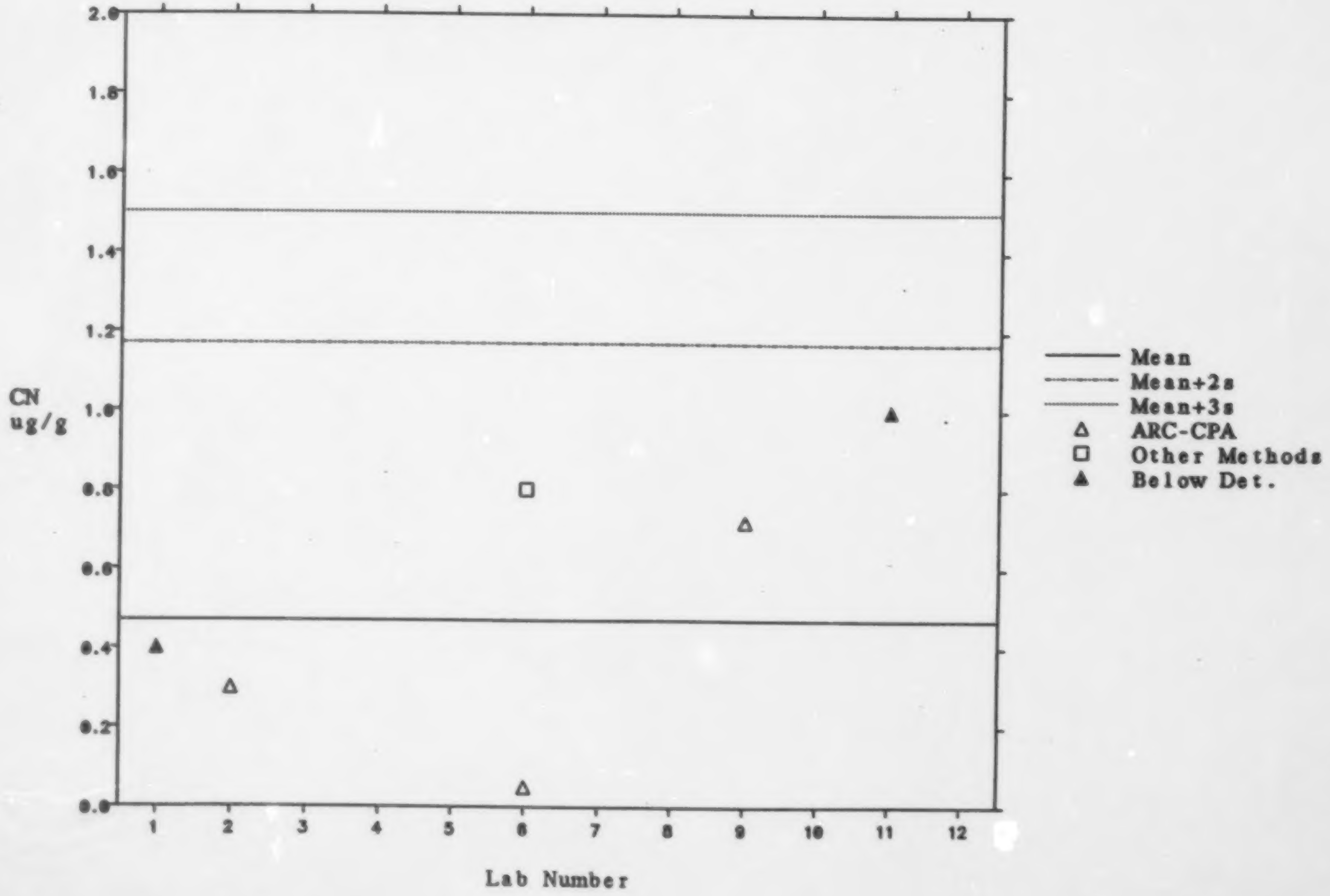


Figure 203. Analysis of CN in Sludge E

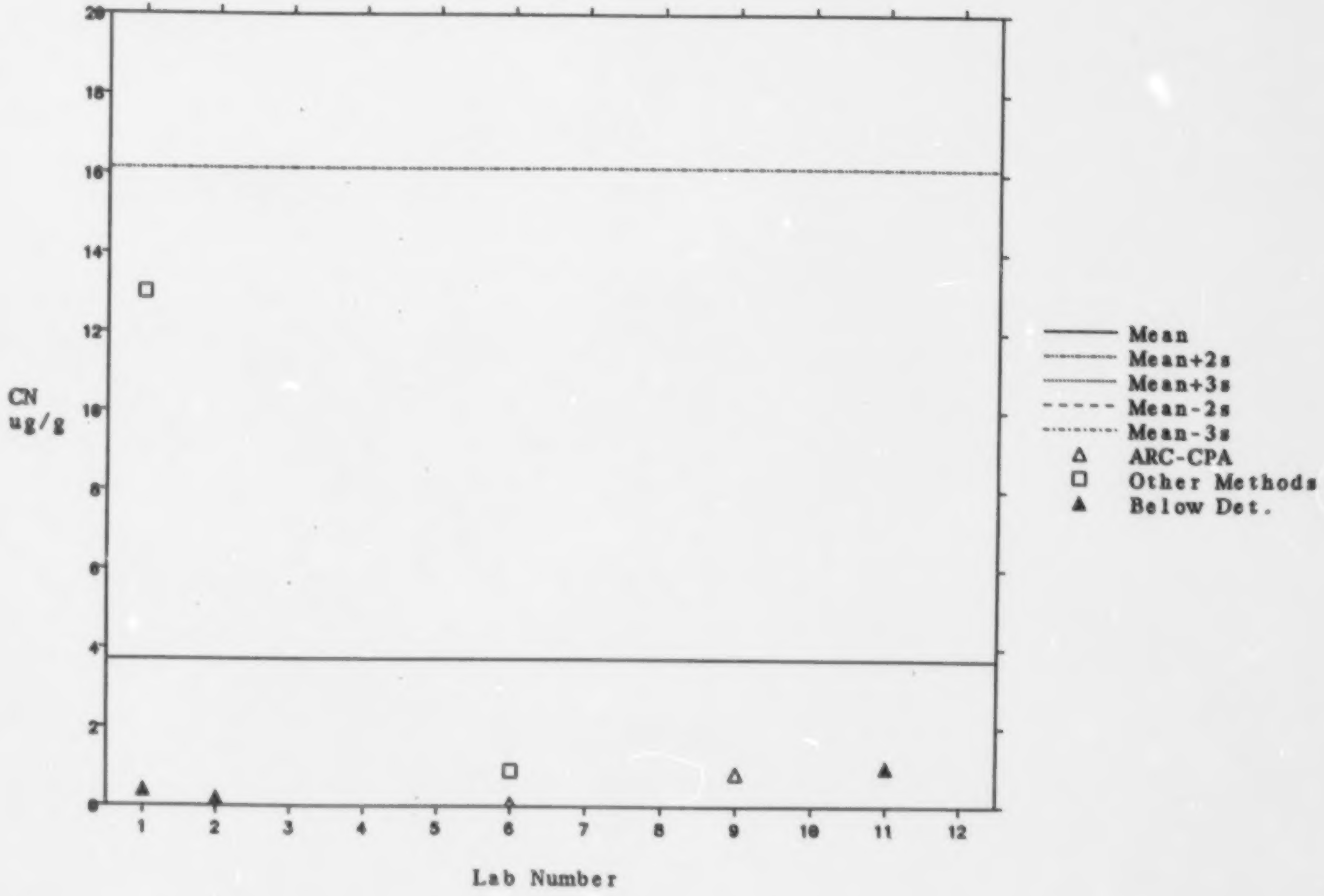
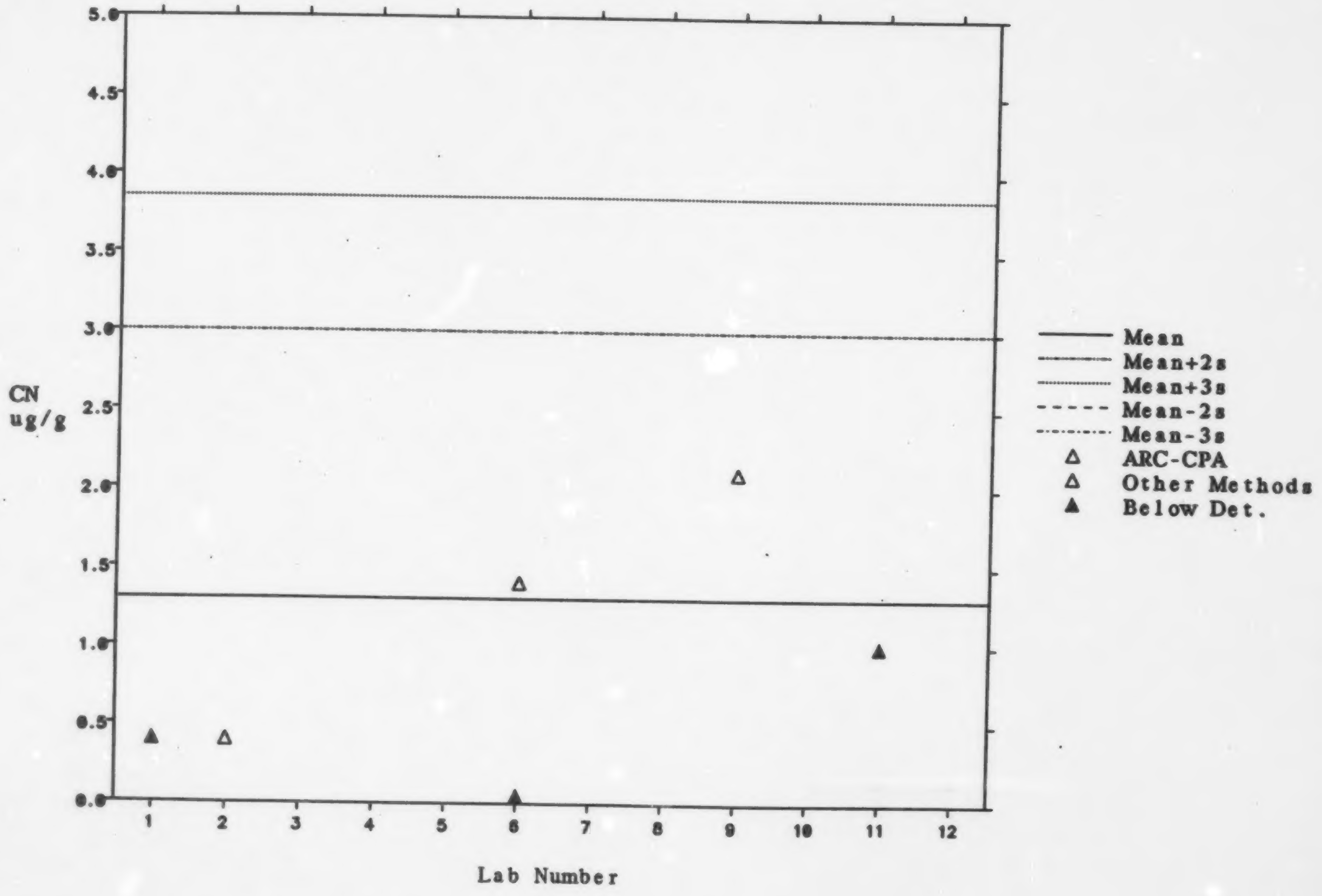


Figure 204. Analysis of CN in Sludge F



Figures 199 to 204 indicate that all the reported CN data fell within the sludges' control limits, with significant differences between the ARC-CPA and some other methods. The CV values and means for CN in several of the sludges would be lowered significantly if results of one lab (using other method) were left out. The lab concerned reported a method that took into account several forms of CN, thereby elevating the reported CN values. If one neglects the high values from the above lab, one can see much better agreement between all labs.

The CN data shows some good agreement between the different labs (except for one) and for the different methodologies used. The variability seems to be partly due to the high values reported by one lab.

5. CONCLUSIONS

The uneven distribution of the reported data for the elements and the varied participation by the laboratories makes it difficult to draw statistically significant conclusions. The observed effects for the different elements could not be confirmed as real differences or artifacts due to the varied methodologies used and/or to the low sample and lab numbers. Based on the discussions in the previous section, we can make the following observational comments only.

The ICP standard solution showed the best recovery of all the tested samples. Most labs were consistently within the standard's control elements. Few labs were outside the control limits and these labs might want to check their results. Based on the ICP data, we believe that most labs are capable and do perform well on standard aqueous samples such as our ICP standard.

The digestate samples did not show as good a recovery as the ICP standard. Digestate 3 showed consistently better results than digestate 4. This could be due to the narrower control limits of digestate 4. Despite some problems, many labs were able to recover most of the elements in these samples. The observed differences between labs and the low recovery of some elements leads us to propose that more attention should be given to the analysis of non homogeneous acid solutions such as the tested digestates.

The standard samples showed poorer results than for the ICP and digestate standards. Problems of sample size, loss of samples and non familiarity of some labs with the material are partly responsible for the reported variation. The variability and recovery pattern exhibited by these samples are due both to the sample digestion step and the ICP analysis procedure (as evidenced from the digestate analysis). Attempts at separating the variability into that due to the digestion or the analysis steps were not possible as the reported sample numbers were not adequate.

The analysis of the 6 sludges showed that some samples contained significant concentrations of heavy metals to be of concern. The recovery of elements from the sludges could not be assessed as the elemental contents are not known accurately. The reproducibility of the data from the labs varied widely for most elements. Coefficient of variability values ranged from acceptable values to more than 200%. This variation was due in most cases to the small number of samples analyzed and/or low elemental concentrations.

Comparison of the methods of digestion showed that in most cases there was no differences between what we termed ARC-CPA method and all other methods used. This seems to be contrary to the results reported by Chow and Abboud (1989) and often mentioned in other literature sources. A closer look showed that labs that used the ARC-CPA method did not follow exactly the methods outlined in Chow and Abboud (1989), rather they used this method as a guideline and did adjust some equipment and extraction conditions as they saw fit. Both the ARC-CPA and "other methods" showed significant variability for most elements and labs, which could be due to the variations in the procedures employed for both methods. The similarity in the performance of the two methods could be a product of the variability of both methods.

6. RECOMMENDATIONS

The results of this study did not answer completely the objectives set out at the start of the project. We feel that the labs were presented with a large number of samples to analyze, study and report; a situation that led many labs to deal with only a small subset of the samples. Unfortunately, the different labs chose different subsets of elements and samples, and thus a patchwork of results was generated that could not answer the questions posed in the study objectives. In order to further assess interlaboratory performance of gas plant sludge analysis, we recommend the following:

1. Small set of samples (and maybe elements) should be carefully chosen and submitted to laboratories for analysis. This is intended to insure acquisition of a complete database for statistical analysis.
2. Larger sample sizes (weights) should be provided, which the labs could subsample for their own analysis.
3. Payment for some labs might be necessary in order to insure proper attention to these samples, which in some cases were not attended to as they were non revenue generating samples.

7. REFERENCES

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Appendix I. Total elemental analysis data for the ICP standard, Digestate standards, certified standards and gas plant sludges A-F.

Table 1. Elemental contents (mg/L) of the ICP standard and the Digestate samples.

Sample	LAB 1	LAB 2	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB 10	LAB 11
				Ag					
Blank		<0.10	<0.010			0.03		0.030	
Dige. 3		2.00	0.050			2.30		2.000	
Dige. 4		2.20	2.250			2.30		2.000	
ICP Std		<0.10	<0.010			<5e-03			
				As					
Blank	0.004		<0.010		0.3180	8.80		<0.050	0.0303
Dige. 3	0.304		0.630		<0.0870	71.00		0.700	1.2500
Dige. 4	0.330		0.780		<0.0870	103.00		0.600	1.3400
ICP Std	<0.100	<0.10	10.000	<0.100	16.1000	2600.00	<0.10	<0.100	9.9200
				B					
Blank									
Dige. 3									
Dige. 4									
ICP Std									
				Ba					
Blank	<0.004	<0.05	0.010	0.220	<0.0080	<5e-03		0.010	0.0073
Dige. 3	7.200	7.80	6.000	9.750	6.4500	7.30		7.200	7.4900
Dige. 4	8.200	9.20	6.480	10.350	7.3900	8.00		8.100	8.3100
ICP Std	9.800	9.40	7.630	9.200	9.4400	8.80			9.9400
				Be					
Blank	<0.003		<0.001					<0.005	<0.0018
Dige. 3	0.009		0.052					0.067	0.0578
Dige. 4	0.010		0.048					0.089	0.0817
ICP Std	9.900		7.420						9.5900
				Cd					
Blank	<0.007	<0.01	0.008	0.020	0.0210	0.02	0.00	0.005	<0.0092
dige. 3	2.550	3.00	1.890	2.750	2.4000	2.20	2.90	2.300	2.5200
dige. 4	2.510	3.00	1.870	2.750	2.3300	2.20	2.80	2.300	2.4800
ICP Std	9.500	10.10	7.530	10.150	9.1800	7.90	11.00		9.7000
				Co					
Blank	<0.007	<0.01	0.011	0.030	0.1010	<0.01	0.00	0.030	<0.0092
dige. 3	0.254	0.14	0.210	0.360	0.3800	0.14	0.15	0.230	0.2890
dige. 4	0.305	0.26	0.260	0.430	0.3800	0.21	0.22	0.290	0.3410
ICP Std	9.800	10.40	7.510	10.250	8.5700	7.80	0.54		10.1000

Table 1. Elemental contents (mg/L) of the ICP standard and the Digestate samples.

Sample	LAB 1	LAB 2	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB 10	LAB 11
				Cr					
Blank	<0.020	<0.01	<0.001	<0.010	0.0360	0.08	0.00	0.020	<0.0183
dige. 3	105.000	118.00	85.000	107.000	99.6000	65.00	99.00	83.000	92.7000
dige. 4	3.340	4.20	3.160	2.950	3.1700	3.50	3.70	3.000	3.3800
ICP Std	9.400	10.80	7.600	9.850	8.6800	9.20	7.50		9.2100
				Cu					
Blank	<0.004	<0.01	0.004	<0.005	<0.0140	<5e-03	0.00	<0.010	0.0110
dige. 3	3.210	3.20	2.480	3.630	3.3700	3.60	3.40	3.500	3.3500
dige. 4	3.050	3.20	2.370	3.600	3.2100	3.20	3.30	3.300	3.1700
ICP Std	9.900	10.50	7.640	10.300	9.7400	9.80	10.10		9.9400
				Hg					
Blank			0.004		0.0520	2.40			
dige. 3			0.440		2.0900	265.00			
dige. 4			0.420		1.8900	230.00			
ICP Std			7.600		0.2700	4.40			
				Mn					
Blank	<0.060	<0.01	0.005	<0.005		4e-03		<0.010	<0.0055
dige. 3	5.600	6.40	4.430	6.250		5.10		3.600	5.6200
dige. 4	3.200	3.80	2.540	3.600		3.20		1.200	3.2200
ICP Std	9.900	10.40	7.600	10.150		8.00			9.8200
				Mo					
Blank		<0.05	<0.005	0.050	<0.0040	<0.01		<0.050	<0.0459
dige. 3		3.20	2.460	3.400	3.3500	2.60		2.000	3.0000
dige. 4		3.20	2.530	3.400	3.4600	2.60		2.000	3.0400
ICP Std		10.60	7.370	10.950	9.2200	8.80			9.8500
				Ni					
Blank	<0.008	<0.10	0.025	0.050	0.1830	<0.01		0.140	<0.0275
dige. 3	3.090	3.30	2.620	3.550	2.9100	2.50		2.700	2.9400
dige. 4	3.140	3.40	2.680	3.560	2.9400	2.60		2.900	3.0200
ICP Std	9.900	10.90	7.930	10.450	8.8800	8.10			9.5800

Table 1. Elemental contents (mg/L) of the ICP standard and the Digestate samples.

Sample	LAB 1	LAB 2	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB 10	LAB 11
Pb									
Blank	<0.040	<0.10	0.020	0.120	<0.3000	0.05	0.00	<0.050	<0.0917
dige. 3	3.970	6.60	4.770	6.000	3.9700	4.40	5.30	4.100	6.2400
dige. 4	1.820	3.80	2.940	3.360	2.1500	2.60	2.90	2.500	4.2400
ICP Std	10.100	10.20	7.970	10.050	7.9500	7.79	10.80		9.8300
Sb									
Blank		<0.50	<0.010			9.80		<0.050	
dige. 3		4.00	1.800			64.00		2.500	
dige. 4		3.00	2.360			350.00		2.100	
ICP Std		<0.50	<0.010			15.00			
Se									
Blank	<2e-04		<0.010		<0.0001	<0.50		0.060	
dige. 3	0.014		<0.010			<0.50		0.300	
dige. 4	0.024		<0.010			<0.50		0.400	
ICP Std			<0.010			<0.50			
Tl									
Blank			<0.010			<0.20		0.100	
dige. 3			3.500			<0.20		6.000	
dige. 4			4.200			<0.20		5.000	
ICP Std			<0.010			<0.20			
V									
Blank	<0.004	<0.10	<0.001	<0.100	<0.0220	8e-03		<0.050	<0.0275
dige. 3	3.730	4.10	3.280	4.300	3.5100	1.20		3.300	3.7300
dige. 4	4.270	4.90	3.800	4.800	4.1000	4.30		3.700	4.2600
ICP Std	9.800	10.00	7.580	7.400	9.6600	9.60			10.4000
Zn									
Blank	<0.009	<0.01	0.045	0.030	0.1370	<5e-03		<0.010	<0.0183
dige. 3	9.200	11.00	7.280	9.850	8.2000	8.20		5.900	9.2000
dige. 4	3.000	4.40	3.040	4.006	3.4900	3.30		3.000	3.7700
ICP Std	10.000	9.90	7.600	10.200	8.8500	7.90			9.8400

Table 2. Elemental concentrations (ug/g) of Standard A and the digestion methods (A=ARC-CPA, O=Other) used.

	Method	Ag	As	B	Ba	Be	Cd	Co (ug/g)	Cr	Cu	Hg	Mn	Mo	Ni	Pb	Tl
LAB 1	A		13.000		410	0.70	128.0	11.80	5000	168		303		152	185	
LAB 1	A		14.000		397	0.80	139.0	12.80	5800	186		301		166	211	
LAB 1	A		14.000		382	0.80	145.0	12.30	5100	189		274		171	200	
LAB 1	A		30.000		194	0.80	128.0	6.10	5400	158		268		130	219	
LAB 1	O		31.000		188	0.80	130.0	6.10	5600	158		270		132	223	
LAB 2	O	190			690		131.0	12.00	9000	220		390	170	212	430	
LAB 2	O	92			760		191.0	12.00	8300	253		460	184	230	350	
LAB 2	A	180			600		187.0	12.00	9800	260		420	200	220	410	
LAB 2	A	180			760		187.0	12.00	9800	230		400	200	220	400	
LAB 2																
LAB 5	A	193	52.000		450	5.00	142.0	22.00	6900	191	22.5	335	219	233	318	117
LAB 5	A	177	53.000		500	4.00	141.0	17.00	6350	187	25.3	325	230	203	290	130
LAB 5	A	180	51.000		550	4.00	142.0	17.00	7130	184	26.9	350	227	220	340	127
LAB 5	A															
LAB 5	A															
LAB 6	A				282		150.0	15.00	5540	190		298	171	186	282	
LAB 6	A				272		144.0	13.00	5230	190		282	171	179	265	
LAB 6	A				284		142.0	13.00	5530	184		284	172	175	278	
LAB 6	A				358		115.0	12.00	4350	146		246	127	139	234	
LAB 6	A				224		116.0	9.00	4000	133		225	123	137	195	
LAB 7	O		<0.087		358		222.0	17.50	7580	272	114.0		215	240	380	
LAB 7	O															
LAB 7	O		<0.087		338		183.0	22.90	6280	204	123.0		209	219	330	
LAB 7	O															
LAB 7	O		<0.087		368		191.0	24.30	7360	219	83.1		205	223	395	
LAB 8	O	129	37.000		420		123.0	6.90	3570	180	8.6	280	134	137	245	<20
LAB 8	O	131	33.000		420		127.0	6.10	3290	183	8.8	280	135	142	250	<20
LAB 8	O	120	34.000		410		112.0	6.00	3440	163	9.9	265	125	122	230	<20
LAB 8	O	122	34.000		405		114.0	6.20	3280	170	12.0	270	117	127	225	<20
LAB 8	O	114	34.000		405		107.0	6.50	3480	154	10.1	255	112	116	225	<20
LAB 9																
LAB 9																
LAB 9																
LAB 9																
LAB 9					360											
LAB 10	A	18	67.000		230	4.00	130.0	12.00	4900	170		180	140	160	250	310
LAB 10	A	97	44.000		290	4.00	120.0	11.00	4700	180		190	92	170	230	270
LAB 10	A	69	50.000		300	2.00	150.0	5.00	5200	160		260	140	160	290	360
LAB 10	A										24.0					
LAB 10	A															
LAB 11	A		79.500		462	3.27	139.0	15.40	5180	187		313	167	161	342	
LAB 11	A		83.300		475	3.26	146.0	15.30	5200	194		321	175	169	343	
LAB 11	A		79.900		458	3.21	135.0	15.30	5320	184		311	161	155	336	
LAB 11	O		40.200	0.0163			40.2	9.99					127		270	184
LAB 11	O		47.100	0.0185			47.1	9.09					145		292	187

Table 2. Elemental concentrations (ug/g) of Standard A and the digestion methods (A=ARC-CPA, O=Other) used.

	V	Zn
	— (ug/g) —	
LAB 1	192	497
LAB 1	207	484
LAB 1	211	441
LAB 1	153	454
LAB 1	155	455
LAB 2	230	700
LAB 2	280	740
LAB 2	290	680
LAB 2	280	690
LAB 2		
LAB 5	216	546
LAB 5	208	525
LAB 5	195	570
LAB 5		
LAB 5		
LAB 6	149	489
LAB 6	149	458
LAB 6	150	475
LAB 6	149	392
LAB 6	142	353
LAB 7	231	706
LAB 7		
LAB 7	219	559
LAB 7		
LAB 7	220	654
LAB 8		430
LAB 8		420
LAB 8		405
LAB 8		400
LAB 8		395
LAB 9		
LAB 9		
LAB 9		
LAB 9		
LAB 9		
LAB 10	190	370
LAB 10	180	340
LAB 10	150	400
LAB 10		
LAB 10		
LAB 11	208	504
LAB 11	216	510
LAB 11	199	512
LAB 11		
LAB 11		

Table 3. Elemental concentrations (ug/g) of Standard B and the digestion methods (A=ARC-CPA, O=Other) used.

	Method	Ag	As	B	Ba	Be	Cd	Co (ug/g)	Cr	Cu	Hg	Mn	Mo	Ni	Pb	Tl
LAB 1	A		15.000		580.0	1.10	140.0	18.1	186	181		195		177	88	
LAB 1	A		15.000		484.0	1.00	125.0	16.5	164	159		170		154	79	
LAB 1	A		14.000		487.0	1.00	133.0	16.2	165	168		179		164	75	
LAB 1	O		40.000		298.0	1.10	150.0	6.9	154	167		172		152	131	
LAB 1	O		33.000		276.0	0.90	136.0	6.9	140	152		156		138	120	
LAB 2	A	200			970.0		126.0	21.0	290	250		270	230	260	260	
LAB 2																
LAB 2	A	200			1000.0		209.0	23.0	300	250		260	250	270	280	
LAB 2	O	210			840.0		225.0	20.0	300	230		265	240	260	300	
LAB 2	O	15			1130.0		170.0	21.0	301	200		260	260	228	250	
LAB 5	A															
LAB 5	A	171	65.000		715.0	6.00	107.0	23.0	246	183	26.1	212	295	208	240	194
LAB 5	A	171	73.000		650.0	7.00	118.0	23.0	240	196	28.8	210	330	210	210	197
LAB 5	A															
LAB 5	A	182	69.000		600.0	6.00	118.0	22.0	244	199	27.4	220	282	210	240	206
LAB 6	A				106.0		155.0	13.0	192	197		152	184	186	133	
LAB 6	A				288.0		150.0	16.0	183	193		173	180	190	157	
LAB 6	A				268.0		140.0	16.0	170	184		156	180	176	154	
LAB 6	A				256.0		148.0	14.0	174	188		162	191	186	156	
LAB 6	A				321.0		150.0	16.0	182	190		176	194	189	161	
LAB 7	O		<0.087		475.0		179.0	15.8	211	195	60.7		195	227	171	
LAB 7	O															
LAB 7	O		<0.087		586.0		206.0	25.5	255	231	102.0		278	274	288	
LAB 7	O															
LAB 7	O		<0.087		597.0		206.0	23.8	277	230	75.1		261	267	246	
LAB 8	O	125	37.000		510.0		108.0	9.5	187	162	7.3	163	128	126	142	<20
LAB 8	O	132	39.000		510.0		118.0	10.8	143	174	9.9	173	130	134	147	<20
LAB 8	O	118	39.000		485.0		112.0	9.3	126	149	9.4	168	124	129	140	<20
LAB 8	O	122	39.000		495.0		117.0	10.1	120	159	10.8	174	130	132	141	<20
LAB 8	O	112	39.000		450.0		117.0	10.1	123	145	9.2	154	125	133	143	<20
LAB 9					694.0											
LAB 9					509.0											
LAB 9					477.0											
LAB 9					451.0											
LAB 9					441.0											
LAB 10	A	120	77.000		140.0	5.00	140.0	16.0	190	170		66	150	180	130	230
LAB 10	A	130	53.000		120.0	5.50	150.0	15.0	210	190		110	120	190	150	200
LAB 10	A	100	49.000		370.0	2.80	150.0	3.0	160	160		150	150	170	160	360
LAB 10	A										14.0					
LAB 10	A															
LAB 11	A		92.280		594.4	4.96	172.0	18.8	227	219		227	211	202	277	
LAB 11	A		97.658		599.6	5.09	144.0	19.7	203	188		198	183	172	262	
LAB 11	A		93.682		590.3	4.95	140.0	19.1	201	184		196	180	167	257	
LAB 11	O		48.493	0.0133			48.5	15.8					137		153	189
LAB 11	O		49.755	0.0146			49.8	14.9					140		136	197

Table 3. Elemental concentrations (ug/g) of Standard B and the digestion methods (A=ARC<CPA, O=Other) used.

	V	Zn
	— (ug/g) —	
LAB 1	258	214
LAB 1	223	188
LAB 1	231	199
LAB 1	185	183
LAB 1	168	165
LAB 2	370	310
LAB 2		
LAB 2	380	320
LAB 2	320	300
LAB 2	350	295
LAB 5		
LAB 5	344	222
LAB 5	332	216
LAB 5		
LAB 5	345	230
LAB 6	224	178
LAB 6	212	199
LAB 6	203	178
LAB 6	207	188
LAB 6	210	194
LAB 7	211	241
LAB 7		
LAB 7	287	280
LAB 7		
LAB 7	291	297
LAB 8	205	171
LAB 8	220	180
LAB 8	215	176
LAB 8	210	166
LAB 8	195	173
LAB 9		
LAB 9		
LAB 9		
LAB 9		
LAB 9		
LAB 10	230	190
LAB 10	230	190
LAB 10	160	180
LAB 10		
LAB 10		
LAB 11	286	251
LAB 11	258	222
LAB 11	254	216
LAB 11		
LAB 11		

Table 4. Silver (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O	<25.0	O	<15.0	O	<7.0	O	<10.0	A	<15	O	<20.0
LAB 5	A	<2.0	A	<2.0	A	<2.0	A	<2.0	A		A	<2.0
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.5	O	<1.0	O	<0.5	O	<0.5	O	<1	O	<0.5
LAB 9	O	2.5	A	2.4			O					
LAB 10	A	<1.0	A	<4.0	A	<2.0	A	<1.0	A	<4	A	<2.0
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	O	<25.0	O	<25.0	A	<7.0	O	<10.0	A	<15	A	<12.0
LAB 5	A		A		A		A		A	<2	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	2.4			O					
LAB 10	A	<1.0	A	<4.0	A	<2.0	A	<1.0	A	<4	A	<2.0
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	A	<7.0	O	<25.0	O	<10.0	O	<10.0	O	<15	A	<12.0
LAB 5	A	<2.0	A		A	<2.0	A		A		A	<2.0
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.5	O	<1.0	O	<0.5	O	<0.5	O	<1	O	<0.5
LAB 9			A	2.5			O					
LAB 10	A	<1.0	A	<4.0	A	<2.0	A	<1.0	A	<4	A	<2.0
LAB 11	A		A		A		A		A		A	
LAB 1	O		O		O		O		O		O	
LAB 2	O	<9.0	A	<15.0	O	<10.0	O	<10.0	O	<15	A	<12.0
LAB 5	A		A	<2.0	A		A	<2.0	A	<2	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	2.1			O					
LAB 10	A	<1.0	A	<4.0	A	<2.0	A	16.0	A	<4	A	<2.0
LAB 11	O		O		O		O		O		O	
LAB 1	O		O		O		O		O		O	
LAB 2	O	<9.0	A	<15.0	A	<7.0	A	<6.0	O	<45	A	<12.0
LAB 5	A	<2.0	A		A	<2.0	A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.5	O	<1.0	O	<0.5	O	<0.5	O	<1	O	<0.5
LAB 9			A	2.0			O	3.0				
LAB 10	A	4.0	A	<4.0	A	<7.0	A	8.0	A	<4	A	5.0
LAB 11	O		O		O		O		O		O	
LAB 1	O		O		O		O		O		O	
LAB 2	A	<7.0	O	<15.0	O	<7.0	A	<6.0	O	<45	A	<20.0
LAB 5	A		A	<2.0	A		A	<2.0	A	<2	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	3.0			O	2.9				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 5. Arsenic (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	1.600	A	4.30	A	1.70	A	3.20	A	<0.02	A	1.70
LAB 2	O		O		O		O		A		O	
LAB 5	A	6.600	A	8.80	A	3.90	A	11.20	A		A	10.20
LAB 6	A		A		A		A		A		A	
LAB 7	O	<0.087	O	<0.09	O	8.41	O	<0.09	O	<0.09	O	<0.09
LAB 8	O	5.200	O	10.00	O	3.70	O	8.40	O	<0.20	O	5.70
LAB 9	O		A			15.00	O					
LAB 10	A	7.000	A	29.00	A	<10.00	A	20.00	A	<20.00	A	28.00
LAB 11	A	7.930	A	37.60	A	15.80	A	17.30	A	<3.94	A	20.90
LAB 1	A	1.600	A	4.10	A	1.50	A	2.30	A	<0.02	A	2.10
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			18.00	O					
LAB 10	A	8.000	A	<20.00	A	<10.00	A	<10.00	A	<20.00	A	17.00
LAB 11	A	6.330	A	36.70	A	15.40	A	13.50	A	<3.44	A	23.10
LAB 1	A	1.600	A	4.40	A	1.60	A	2.30	A	0.03	A	2.10
LAB 2	A		O		O		O		O		A	
LAB 5	A	5.900	A		A	3.80	A		A		A	9.80
LAB 6	A		A		A		A		A		A	
LAB 7	O	<0.087	O	<0.09	O	2.32	O	<0.09	O	16.40	O	<0.09
LAB 8	O	5.000	O	9.90	O	3.90	O	8.30	O	<0.20	O	4.80
LAB 9			A			14.00	O					
LAB 10	A	13.000	A	<20.00	A	<10.00	A	<10.00	A	<20.00	A	<10.00
LAB 11	A	5.860	A	38.20	A	14.50	A	13.10	A	4.75	A	
LAB 1	O	3.300	O	8.00	O	3.20	O	6.00	O	0.06	O	6.00
LAB 2	O		A		O		O		O		A	
LAB 5	A		A	7.70	A		A	9.60	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O	<0.09	O		O		O	<0.09	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			9.10	O					
LAB 10	A	6.000	A	24.00	A	<10.00	A	14.00	A	<20.00	A	<10.00
LAB 11	O	1.800	O	20.90	O	3.06	O	13.30	O	2.03	O	15.00
LAB 1	O	2.900	O	7.00	O	3.30	O	6.00	O	0.06	O	6.00
LAB 2	O		A		A		A		O		A	
LAB 5	A	7.200	A		A	3.60	A		A		A	10.00
LAB 6	A		A		A		A		A		A	
LAB 7	O	<0.087	O		O	8.83	O	<0.09	O		O	<0.09
LAB 8	O	5.000	O	10.00	O	3.90	O	8.90	O	<0.20	O	5.60
LAB 9			A			14.00	O					
LAB 10	A	5.000	A	<20.00	A	<10.00	A	19.00	A	<20.00	A	<10.00
LAB 11	O	2.910	O	23.00	O	4.44	O	10.10	O	<2.44	O	14.80
LAB 1	O	3.400	O	7.00	O	3.00	O	6.00	O	0.07	O	5.00
LAB 2	A		O		O		A		O		A	
LAB 5	A		A	7.60	A		A	10.80	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			3.10	O					
LAB 10	A		A		A		A		A		A	<10.00
LAB 11	O	1.990	O	23.50	O	0.60	O	10.80	O	<2.59	O	17.00

Table 6. Boron (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		O		O		A		O	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	33.000	O	146.00	O	13.000	O	38.000	O	5.800	O	60.000
LAB 9	O		A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	A		O		O		O		O		A	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	48.000	O	144.00	O	14.000	O	38.000	O	5.200	O	62.000
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		O		O		O		A	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.010	O	5e-03	O	0.003	O		O	0.005	O	0.004
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		A		A		O		A	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	43.000	O	147.00	O	15.000	O	40.000	O	4.700	O	61.000
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.010	O	5e-03	O	<0.003	O	0.003	O	<0.005	O	0.004
LAB 1	O		O		O		O		O		O	
LAB 2	A		O		O		A		O		A	
LAB 5	A		A		A		A		A		A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.009	O	<5e-03	O	<0.003	O	0.004	O	<0.005	O	0.004

Table 7. Barium (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	107	A	910	A	429	A	364	A	<0.20	A	710
LAB 2	O	130	O	1060	O	503	O	697	A	<8.00	O	1800
LAB 5	A	204	A	820	A	473	A	520	A		A	1450
LAB 6	A	930	A	1120	A	1790	A	725	A	<4.00	A	505
LAB 7	O	141	O	561	O	404	O	583	O	<0.04	O	605
LAB 8	O	133	O	940	O	440	O	520	O	0.80	O	290
LAB 9	O	252	A	1000		325	O					
LAB 10	A	120	A	940	A	140	A	310	A	70.00	A	1000
LAB 11	A	139	A	920	A	443	A	419	A	<0.66	A	1040
LAB 1	A	105	A	910	A	447	A	331	A	<0.20	A	680
LAB 2	O	120	O	1010	A	420	O	660	A	<8.00	A	963
LAB 5	A		A		A		A		A	<1.00	A	
LAB 6	A	680	A	945	A	1790	A	750	A	<4.00	A	445
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	1020		309	O					
LAB 10	A	130	A	880	A	360	A	380	A	8.00	A	850
LAB 11	A	139	A	919	A	483	A	476	A	<0.57	A	1000
LAB 1	A	103	A	910	A	440	A	362	A	<0.20	A	650
LAB 2	A	140	O	960	O	390	O	590	O	<8.00	A	926
LAB 5	A	210	A		A	482	A		A		A	1420
LAB 6	A	779	A	980	A	1790	A	700	A	<4.00	A	515
LAB 7	O	126	O	616	O	396	O	631	O	0.40	O	681
LAB 8	O	131	O	950	O	450	O	480	O	0.60	O	77
LAB 9			A	1090		311	O					
LAB 10	A	130	A	910	A	370	A	420	A	6.00	A	870
LAB 11	A	139	A	972	A	495	A	451	A	<0.59	A	
LAB 1	O	69	O	500	O	396	O	306	O	<0.20	O	256
LAB 2	O	170	A	1120	O	390	O	670	O	<8.00	A	1300
LAB 5	A		A	880	A		A	500	A	<1.00	A	
LAB 6	A	804	A	1030	A	1780	A	670	A	<4.00	A	480
LAB 7	O		O	641	O		O		O	0.45	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	984		295	O					
LAB 10	A	91	A	640	A	410	A	500	A	8.00	A	1100
LAB 11	O		O		O		O	426	O		O	
LAB 1	O	69	O	520	O	394	O	306	O	<0.20	O	179
LAB 2	O	160	A	1180	A	420	A	430	O	<45.00	A	1600
LAB 5	A	215	A		A	460	A		A		A	1500
LAB 6	A	767	A	1010	A	1780	A	730	A	<4.00	A	550
LAB 7	O	149	O		O	406	O	487	O		O	1300
LAB 8	O	130	O	900	O	450	O	235	O	0.60	O	90
LAB 9			A	1120		326	O	712				
LAB 10	A	92	A	570	A	420	A	530	A	4.00	A	1100
LAB 11	O		O		O		O		O		O	
LAB 1	O	69	O	500	O	392	O	285	O	<0.20	O	225
LAB 2	A	110	O	1000	O	512	A	480	O	<45.00	A	1800
LAB 5	A		A	860	A		A	490	A	<1.00	A	
LAB 6	A	766	A	1020	A	1760	A	750	A	<4.00	A	525
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	1100		347	O	642				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 8. Beryllium (mg/kg) in sludges A–F and the digestion methods (A= ARC–CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	<0.100	A	<0.100	A	<0.100	A	<0.100	A	0.100	A	<0.100
LAB 2	O		O		O		O		A		O	
LAB 5	A	<1.000	A	<1.000	A	<1.000	A	<1.000	A		A	3.000
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.100	O	<0.200	O	<0.100	O	<0.100	O	<0.200	O	<0.100
LAB 9	O		A				O					
LAB 10	A	1.000	A	4.000	A	<1.000	A	<0.500	A	<2.000	A	<1.000
LAB 11	A	0.396	A	0.903	A	<0.141	A	0.102	A	<0.263	A	0.349
LAB 1	A	<0.100	A	<0.100	A	<0.100	A	<0.100	A	0.100	A	<0.100
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A	<1.000	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<0.500	A	<2.000	A	<1.000	A	<0.500	A	<2.000	A	1.000
LAB 11	A	0.288	A	0.885	A	<0.135	A	<0.096	A	<0.229	A	0.372
LAB 1	A	<0.100	A	<0.100	A	<0.100	A	<0.100	A	<0.100	A	<0.100
LAB 2	A		O		O		O		O		A	
LAB 5	A	<1.000	A		A	<1.000	A		A		A	3.000
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.100	O	<0.200	O	<0.100	O	<0.100	O	<0.200	O	<0.100
LAB 9			A				O					
LAB 10	A	0.600	A	<2.000	A	<1.000	A	0.600	A	<2.000	A	<1.000
LAB 11	A	0.249	A	0.828	A	<0.132	A	0.091	A	<0.238	A	
LAB 1	O	0.100	O	0.300	O	0.100	O	<0.100	O	<0.100	O	<0.100
LAB 2	O		A		O		O		O		A	
LAB 5	A		A	<1.000	A		A	<1.000	A	<1.000	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	0.600	A	<2.000	A	<1.000	A	0.500	A	<2.000	A	<1.000
LAB 11	O		O		O		O	0.138	O		O	
LAB 1	O	0.100	O	0.200	O	<0.100	O	<0.100	O	<0.100	O	<0.100
LAB 2	O		A		A		A		O		A	
LAB 5	A	<1.000	A		A	<1.000	A		A		A	3.000
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<0.100	O	<0.200	O	<0.100	O	<0.100	O	<0.200	O	<0.100
LAB 9			A				O					
LAB 10	A	0.500	A	<2.000	A	<1.000	A	<0.500	A	<2.000	A	<1.000
LAB 11	O		O		O		O		O		O	
LAB 1	O	0.100	O	0.200	O	<0.100	O	<0.100	O	<0.100	O	<0.100
LAB 2	A		O		O		A		O		A	
LAB 5	A		A	<1.000	A		A	<1.000	A	<1.000	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 9. Cadmium (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	<0.400	A	<0.40	A	1.300	A	0.700	A	<0.400	A	1.30
LAB 2	O	1.000	O	<1.00	O	<1.000	O	2.000	A	<1.000	O	1.00
LAB 5	A	<1.000	A	<1.00	A	<1.000	A	<1.000	A		A	<1.00
LAB 6	A	2.500	A	6.00	A	6.000	A	2.500	A	<2.000	A	2.00
LAB 7	O	18.800	O	14.70	O	34.000	O	9.190	O	2.190	O	12.20
LAB 8	O	<0.500	O	<1.00	O	<0.500	O	<0.500	O	<1.000	O	<0.50
LAB 9	O		A				O					
LAB 10	A	<0.500	A	<2.00	A	1.000	A	0.900	A	<2.000	A	2.00
LAB 11	A	<0.396	A	10.80	A	0.706	A	2.200	A	<1.310	A	32.10
LAB 1	A	<0.400	A	<0.40	A	1.000	A	1.000	A	<0.400	A	0.40
LAB 2	O	<1.000	O	2.00	A	<1.000	O	2.000	A	<1.000	A	<1.00
LAB 5	A		A		A		A		A	<1.000	A	
LAB 6	A	2.000	A	4.50	A	6.500	A	3.000	A	<2.000	A	2.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<0.500	A	8.00	A	<1.000	A	0.500	A	<2.000	A	<1.00
LAB 11	A	<0.411	A	8.60	A	<0.674	A	1.480	A	<1.150	A	35.70
LAB 1	A	<0.400	A	<0.40	A	0.800	A	1.000	A	<0.400	A	1.30
LAB 2	A	<1.000	O	2.00	O	<1.000	O	1.000	O	<1.000	A	<1.00
LAB 5	A	<1.000	A		A	<1.000	A		A		A	<1.00
LAB 6	A	2.000	A	5.50	A	6.500	A	3.000	A	<2.000	A	2.00
LAB 7	O	12.200	O	15.20	O	29.600	O	8.190	O	1.600	O	13.60
LAB 8	O	<0.500	O	<1.00	O	<0.500	O	<0.500	O	<1.000	O	<0.50
LAB 9			A				O					
LAB 10	A	<0.500	A	<2.00	A	<1.000	A	<0.500	A	<2.000	A	<1.00
LAB 11	A	<0.416	A	9.24	A	<0.661	A	1.410	A	<1.190	A	
LAB 1	O	<0.400	O	3.60	O	1.000	O	0.300	O	0.600	O	1.50
LAB 2	O	<1.000	A	<1.00	O	<1.000	O	1.000	O	<1.000	A	2.00
LAB 5	A		A	<1.00	A		A	<1.000	A	<1.000	A	
LAB 6	A	2.500	A	6.00	A	6.000	A	2.500	A	<2.000	A	2.00
LAB 7	O		O	11.70	O		O		O	2.230	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<0.500	A	4.00	A	<1.000	A	0.800	A	4.000	A	15.00
LAB 11	O	0.280	O	16.70	O	0.816	O	2.670	O	0.358	O	2.14
LAB 1	O	<0.400	O	2.10	O	0.400	O	43.600	O	<0.400	O	2.20
LAB 2	O	<1.000	A	<1.00	A	<1.000	A	<1.000	O	<1.000	A	2.00
LAB 5	A	<1.000	A		A	<1.000	A		A		A	<1.00
LAB 6	A	2.000	A	6.50	A	6.000	A	3.000	A	<2.000	A	2.00
LAB 7	O	14.900	O		O	35.700	O	9.270	O		O	13.60
LAB 8	O	<0.500	O	<1.00	O	<0.500	O	<0.500	O	<1.000	O	<0.50
LAB 9			A				O					
LAB 10	A	<0.500	A	5.00	A	<1.000	A	3.600	A	19.000	A	<1.00
LAB 11	O	0.166	O	7.52	O	0.403	O	0.046	O	<0.122	O	2.22
LAB 1	O	<0.400	O	2.40	O	0.400	O	0.600	O	<0.400	O	1.20
LAB 2	A	<1.000	O	<1.00	O	<1.000	A	<1.000	O	<1.000	A	1.00
LAB 5	A		A	<1.00	A		A	<1.000	A	<1.000	A	
LAB 6	A	2.000	A	6.50	A	6.000	A	2.500	A	<2.000	A	2.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.192	O	6.59	O	0.537	O	<0.133	O	<0.588	O	2.21

Table 10. Cobalt (mg/kg) in sludges A–F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	2.000	A	55.0	A	3.500	A	14.5	A	<0.30	A	5.90
LAB 2	O	4.000	O	56.0	O	<1.000	O	28.0	A	<2.00	O	8.00
LAB 5	A	4.000	A	53.0	A	1.000	A	27.0	A		A	9.00
LAB 6	A	9.000	A	74.0	A	18.000	A	22.0	A	<4.00	A	11.00
LAB 7	O	31.400	O	78.5	O	37.100	O	35.0	O	4.39	O	22.80
LAB 8	O	2.300	O	58.0	O	2.900	O	15.0	O	<2.00	O	11.00
LAB 9	O		A	65.0			O	24.0				
LAB 10	A	3.000	A	60.0	A	3.000	A	14.0	A	<12.00	A	7.00
LAB 11	A	<0.396	A	61.7	A	<0.706	A	14.4	A	<1.31	A	4.61
LAB 1	A	2.400	A	58.0	A	3.400	A	14.7	A	<0.30	A	5.90
LAB 2	O	2.000	O	60.0	A	<0.800	O	31.0	A	<2.00	A	2.00
LAB 5	A		A		A		A		A	<1.00	A	
LAB 6	A	7.000	A	72.0	A	15.000	A	22.0	A	<4.00	A	10.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	63.0			O	23.0				
LAB 10	A	<3.000	A	69.0	A	3.000	A	17.0	A	<12.00	A	<6.00
LAB 11	A	<0.411	A	61.3	A	<0.674	A	14.0	A	<1.15	A	5.51
LAB 1	A	2.400	A	60.0	A	3.400	A	14.6	A	<0.30	A	5.30
LAB 2	A	1.000	O	60.0	O	2.000	O	20.0	O	<2.00	A	<1.00
LAB 5	A	3.000	A		A	3.000	A		A		A	10.00
LAB 6	A	6.000	A	71.0	A	15.000	A	21.0	A	<4.00	A	11.00
LAB 7	O	7.900	O	90.7	O	32.500	O	32.4	O	4.40	O	30.40
LAB 8	O	2.700	O	58.0	O	2.400	O	15.9	O	<2.00	O	10.00
LAB 9			A	68.0			O	24.0				
LAB 10	A	<3.000	A	68.0	A	3.000	A	15.0	A	<12.00	A	6.00
LAB 11	A	<0.416	A	62.9	A	<0.661	A	13.3	A	<1.19	A	
LAB 1	O	1.200	O	55.0	O	2.900	O	12.8	O	<0.30	O	5.50
LAB 2	O	<1.000	A	63.0	O	2.000	O	27.0	O	<2.00	A	<1.00
LAB 5	A		A	52.0	A		A	20.0	A	<1.00	A	
LAB 6	A	10.000	A	75.0	A	18.000	A	21.0	A	<4.00	A	13.00
LAB 7	O		O	78.7	O		O		O	4.46	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	68.0			O	27.0				
LAB 10	A	<3.000	A	59.0	A	3.000	A	16.0	A	<12.00	A	7.00
LAB 11	O	3.040	O	47.0	O	1.770	O	15.3	O	2.03	O	8.28
LAB 1	O	1.000	O	55.0	O	2.400	O	13.4	O	0.60	O	5.50
LAB 2	O	<1.000	A	64.0	A	<0.800	A	17.0	O	<5.00	A	<1.00
LAB 5	A	4.000	A		A	3.000	A		A		A	11.00
LAB 6	A	9.000	A	75.0	A	16.000	A	21.0	A	<4.00	A	11.00
LAB 7	O	17.400	O		O	37.800	O	26.6	O		O	28.00
LAB 8	O	2.300	O	55.0	O	2.400	O	15.6	O	<2.00	O	10.50
LAB 9			A	64.0			O	22.0				
LAB 10	A	<3.000	A	58.0	A	<3.000	A	16.0	A	<12.00	A	6.00
LAB 11	O	3.070	O	48.3	O	0.941	O	20.7	O	<0.37	O	6.58
LAB 1	O	1.300	O	55.0	O	2.800	O	13.4	O	0.80	O	5.50
LAB 2	A	1.000	O	62.0	O	<1.000	A	20.0	O	<5.00	A	6.00
LAB 5	A		A	54.0	A		A	20.0	A	<1.00	A	
LAB 6	A	7.000	A	76.0	A	18.000	A	21.0	A	<4.00	A	11.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	64.0			O	24.0				
LAB 10	A		A		A		A		A		A	
LAB 11	O	2.030	O	56.8	O	1.140	O	16.6	O	<0.24	O	7.11

Table 11. Chromium (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	18.00	A	232	A	36.0	A	58.0	A	123	A	22.0
LAB 2	O	29.00	O	330	O	42.0	O	150.0	A	147	O	51.0
LAB 5	A	39.00	A	278	A	50.0	A	110.0	A		A	80.0
LAB 6	A	31.00	A	226	A	48.0	A	70.0	A	139	A	26.0
LAB 7	O	60.40	O	297	O	45.5	O	86.6	O	118	O	42.9
LAB 8	O	30.00	O	245	O	54.0	O	125.0	O	179	O	55.0
LAB 9	O	62.00	A	372			O	153.0				
LAB 10	A	17.00	A	260	A	10.0	A	83.0	A	120	A	34.0
LAB 11	A	19.70	A	270	A	37.5	A	93.1	A	119	A	29.2
LAB 1	A	19.00	A	241	A	36.0	A	59.0	A	123	A	23.0
LAB 2	O	29.00	O	300	A	38.0	O	140.0	A	141	A	47.0
LAB 5	A		A		A		A		A	114	A	
LAB 6	A	21.00	A	213	A	49.0	A	68.0	A	143	A	27.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	336			O	135.0				
LAB 10	A	24.00	A	260	A	24.0	A	77.0	A	130	A	22.0
LAB 11	A	19.30	A	267	A	39.2	A	88.5	A	112	A	32.1
LAB 1	A	19.00	A	256	A	34.0	A	81.0	A	129	A	23.0
LAB 2	A	25.00	O	320	O	40.0	O	150.0	O	197	A	51.0
LAB 5	A	33.00	A		A	58.0	A		A		A	82.0
LAB 6	A	27.00	A	233	A	48.0	A	68.0	A	133	A	29.0
LAB 7	O	0.64	O	314	O	40.0	O	92.9	O	153	O	47.5
LAB 8	O	34.00	O	210	O	57.0	O	110.0	O	167	O	52.0
LAB 9			A	402			O	171.0				
LAB 10	A	22.00	A	260	A	25.0	A	73.0	A	120	A	25.0
LAB 11	A	19.50	A	283	A	39.3	A	97.9	A	111	A	
LAB 1	O	16.00	O	242	O	31.0	O	42.0	O	124	O	19.0
LAB 2	O	36.00	A	320	O	39.0	O	200.0	O	130	A	61.0
LAB 5	A		A	278	A		A	105.0	A	117	A	
LAB 6	A	27.00	A	229	A	48.0	A	62.0	A	135	A	27.0
LAB 7	O		O	289	O		O		O	139	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	333			O	214.0				
LAB 10	A	16.00	A	240	A	33.0	A	56.0	A	130	A	17.0
LAB 11	O		O		O		O	88.9	O		O	
LAB 1	O	16.00	O	244	O	31.0	O	42.0	O	122	O	18.0
LAB 2	O	31.00	A	300	A	40.0	A	76.0	O	125	A	63.0
LAB 5	A	44.00	A		A	49.0	A		A		A	77.0
LAB 6	A	27.00	A	238	A	49.0	A	66.0	A	137	A	27.0
LAB 7	O	34.10	O		O	45.0	O	72.1	O		O	38.1
LAB 8	O	38.00	O	265	O	58.0	O	58.0	O	172	O	64.0
LAB 9			A	330			O	157.0				
LAB 10	A	16.00	A	230	A	32.0	A	67.0	A	120	A	17.0
LAB 11	O		O		O		O		O		O	
LAB 1	O	16.00	O	252	O	32.0	O	42.0	O	120	O	17.0
LAB 2	A	24.00	O	320	O	42.0	A	85.0	O	119	A	54.0
LAB 5	A		A	277	A		A	110.0	A	119	A	
LAB 6	A	28.00	A	227	A	49.0	A	71.0	A	144	A	28.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	322			O	162.0				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 12. Copper (mg/kg) in sludges A–F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	9.50	A	234	A	53.0	A	32.8	A	<0.200	A	31.5
LAB 2	O	12.00	O	290	O	62.0	O	69.0	A	<2.000	O	51.0
LAB 5	A	23.00	A	212	A	56.0	A	42.0	A		A	58.0
LAB 6	A	16.00	A	265	A	66.0	A	43.0	A	<2.000	A	40.0
LAB 7	O	21.70	O	268	O	71.1	O	52.9	O	<0.063	O	62.4
LAB 8	O	9.20	O	210	O	40.0	O	29.0	O		O	36.0
LAB 9	O	24.00	A	287			O	63.0				
LAB 10	A	13.00	A	250	A	57.0	A	34.0	A	<4.000	A	36.0
LAB 11	A	6.58	A	248	A	55.0	A	41.9	A	<1.310	A	29.5
LAB 1	A	9.00	A	248	A	56.0	A	28.2	A	<0.200	A	31.3
LAB 2	O	11.00	O	240	A	50.0	O	67.0	A	<2.000	A	32.0
LAB 5	A		A		A		A		A	<1.000	A	
LAB 6	A	10.50	A	251	A	72.0	A	47.0	A	<2.000	A	39.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	293			O	54.0				
LAB 10	A	13.00	A	260	A	58.0	A	34.0	A	5.000	A	31.0
LAB 11	A	5.72	A	245	A	59.3	A	34.5	A	<1.150	A	33.0
LAB 1	A	9.10	A	245	A	53.0	A	30.2	A	<0.200	A	36.8
LAB 2	A	2.00	O	250	O	55.0	O	40.0	O	<2.000	A	35.0
LAB 5	A	14.00	A		A	50.0	A		A		A	59.0
LAB 6	A	13.50	A	274	A	67.0	A	44.0	A	<2.000	A	43.0
LAB 7	O	3.84	O	291	O	68.4	O	38.1	O	<0.063	O	63.4
LAB 8	O	8.10	O	205	O	39.0	O	32.0	O		O	47.0
LAB 9			A	304			O	54.0				
LAB 10	A	11.00	A	250	A	58.0	A	34.0	A	4.000	A	37.0
LAB 11	A	5.53	A	247	A	58.7	A	31.6	A	<1.190	A	
LAB 1	O	8.10	O	253	O	48.5	O	23.6	O	<0.200	O	36.9
LAB 2	O	16.00	A	260	O	57.0	O	50.0	O	<2.000	A	64.0
LAB 5	A		A	210	A		A	39.0	A	<1.000	A	
LAB 6	A	14.50	A	273	A	68.0	A	44.0	A	<2.000	A	41.0
LAB 7	O		O	276	O		O		O	<0.063	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	289			O	72.0				
LAB 10	A	13.00	A	330	A	57.0	A	32.0	A	11.000	A	39.0
LAB 11	O		O		O		O	48.7	O		O	
LAB 1	O	8.40	O	247	O	49.5	O	24.2	O	<0.200	O	34.4
LAB 2	O	13.00	A	260	A	50.0	A	27.0	O	<5.000	A	92.0
LAB 5	A	8.00	A		A	58.0	A		A		A	58.0
LAB 6	A	13.50	A	267	A	66.0	A	45.0	A	<2.000	A	40.0
LAB 7	O	25.10	O		O	70.6	O	40.9	O		O	58.7
LAB 8	O	6.80	O	210	O	39.0	O	42.0	O		O	41.0
LAB 9			A	285			O	87.0				
LAB 10	A	13.00	A	250	A	68.0	A	31.0	A	10.000	A	37.0
LAB 11	O		O		O		O		O		O	
LAB 1	O	8.10	O	258	O	50.0	O	23.2	O	<0.200	O	33.8
LAB 2	A	3.00	O	280	O	59.0	A	27.0	O	<5.000	A	46.0
LAB 5	A		A	220	A		A	37.0	A	<1.000	A	
LAB 6	A	14.00	A	269	A	68.0	A	46.0	A	<2.000	A	39.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	295			O	56.0				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 13. Mercury (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		O		O		A		O	
LAB 5	A	1.44	A	26.8	A	2.30	A	2.93	A		A	2.75
LAB 6	A		A		A		A		A		A	
LAB 7	O	22.70	O	206.0	O	17.20	O	65.20	O	<0.065	O	166.00
LAB 8	O	0.32	O	11.0	O	0.55	O	0.27	O	0.020	O	0.30
LAB 9	O		A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A	0.250	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	A		O		O		O		O		A	
LAB 5	A	1.14	A		A	2.14	A		A		A	3.36
LAB 6	A		A		A		A		A		A	
LAB 7	O	1.50	O	131.0	O	15.40	O	61.70	O	<0.065	O	76.70
LAB 8	O	0.34	O	8.3	O	0.23	O	0.22	O	0.040	O	0.43
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	A		A		A		A		A		A	
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		O		O		O		A	
LAB 5	A		A	24.6	A		A	2.80	A	0.320	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O	236.0	O		O		O	0.450	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		A		A		O		A	
LAB 5	A	1.39	A		A	2.28	A		A		A	2.89
LAB 6	A		A		A		A		A		A	
LAB 7	O	27.20	O		O	16.00	O	47.20	O		O	65.20
LAB 8	O	0.12	O	10.8	O	0.23	O	0.54	O	0.020	O	0.08
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	
LAB 1	O		O		O		O		O		O	
LAB 2	A		O		O		A		O		A	
LAB 5	A		A	24.6	A		A	2.93	A	0.270	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	0.50	A	0.4	A	0.40	A	0.80	A	<0.100	A	1.60
LAB 11	O		O		O		O		O		O	

Table 14. Manganese (mg/kg) in sludges A–F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	73.0	A	640	A	97	A	85	A	7.00	A	162
LAB 2	O	100.0	O	700	O	113	O	167	A	8.00	O	303
LAB 5	A	138.0	A	535	A	99	A	127	A		A	270
LAB 6	A	113.0	A	607	A	127	A	86	A	9.50	A	195
LAB 7	O		O		O		O		O		O	
LAB 8	O	88.0	O	700	O	103	O	99	O	8.00	O	186
LAB 9	O	119.0	A	652			O					
LAB 10	A	64.0	A	470	A	68	A	99	A	<4.00	A	100
LAB 11	A	83.3	A	641	A	102	A	143	A	5.92	A	181
LAB 1	A	71.0	A	620	A	100	A	82	A	7.00	A	161
LAB 2	O	100.0	O	610	A	108	O	161	A	8.00	A	290
LAB 5	A		A		A		A		A	4.00	A	
LAB 6	A	83.0	A	592	A	124	A	96	A	9.00	A	190
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	632			O					
LAB 10	A	64.0	A	470	A	67	A	84	A	16.00	A	100
LAB 11	A	84.8	A	614	A	108	A	147	A	6.77	A	188
LAB 1	A	72.0	A	600	A	98	A	84	A	7.00	A	162
LAB 2	A	90.0	O	660	O	100	O	104	O	10.00	A	296
LAB 5	A	140.0	A		A	95	A		A		A	310
LAB 6	A	99.0	A	623	A	127	A	81	A	7.50	A	202
LAB 7	O		O		O		O		O		O	
LAB 8	O	89.0	O	640	O	104	O	88	O	8.40	O	190
LAB 9			A	618			O					
LAB 10	A	62.0	A	500	A	68	A	92	A	8.00	A	120
LAB 11	A	86.1	A	619	A	111	A	142	A	5.94	A	
LAB 1	O	67.0	O	570	O	90	O	51	O	7.00	O	179
LAB 2	O	130.0	A	660	O	100	O	120	O	7.00	A	350
LAB 5	A		A	545	A		A	99	A	7.00	A	
LAB 6	A	99.0	A	604	A	127	A	82	A	7.00	A	203
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	580			O					
LAB 10	A	86.0	A	590	A	72	A	110	A	8.00	A	150
LAB 11	O		O		O		O	141	O		O	
LAB 1	O	66.0	O	560	O	92	O	57	O	6.00	O	171
LAB 2	O	106.0	A	740	A	103	A	80	O	8.00	A	420
LAB 5	A	143.0	A		A	96	A		A		A	330
LAB 6	A	97.0	A	621	A	127	A	91	A	7.00	A	200
LAB 7	O		O		O		O		O		O	
LAB 8	O	90.0	O	665	O	107	O	101	O	9.20	O	190
LAB 9			A	627			O	147				
LAB 10	A	87.0	A	570	A	69	A	110	A	7.00	A	160
LAB 11	O		O		O		O		O		O	
LAB 1	O	67.0	O	570	O	92	O	47	O	6.00	O	169
LAB 2	A	90.0	O	700	O	118	A	90	O	4.00	A	321
LAB 5	A		A	580	A		A	194	A	6.00	A	
LAB 6	A	100.0	A	630	A	126	A	93	A	7.50	A	200
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	635			O	189				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 15. Molybdenum (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O	6.00	O	4.00	O	10.00	O	12.00	A	12.00	O	9.00
LAB 5	A	14.00	A	<2.00	A	6.00	A	9.00	A		A	5.00
LAB 6	A	13.00	A	19.00	A	10.00	A	7.00	A	<20.00	A	<10.00
LAB 7	O	63.80	O	68.20	O	138.00	O	19.70	O	14.00	O	86.80
LAB 8	O	<1.00	O	<2.00	O	<1.00	O	<1.00	O	35.00	O	17.50
LAB 9	O		A				O					
LAB 10	A	<5.00	A	<20.00	A	<10.00	A	6.00	A	<20.00	A	<10.00
LAB 11	A	4.76	A	21.70	A	10.60	A	5.21	A	<6.57	A	10.80
LAB 1	A		A		A		A		A		A	
LAB 2	O	7.00	O	12.00	A	6.00	O	15.00	A	10.00	A	10.00
LAB 5	A		A		A		A		A	<2.00	A	
LAB 6	A	12.00	A	21.00	A	11.00	A	<7.00	A	<20.00	A	<10.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.00	A	<10.00	A	<5.00	A	<20.00	A	<10.00
LAB 11	A	3.74	A	21.70	A	9.71	A	6.74	A	8.26	A	12.90
LAB 1	A		A		A		A		A		A	
LAB 2	A	5.00	O	10.00	O	10.00	O	9.00	O	15.00	A	10.00
LAB 5	A	10.00	A		A	8.00	A		A		A	5.00
LAB 6	A	12.00	A	19.00	A	10.00	A	7.00	A	<20.00	A	<10.00
LAB 7	O	7.48	O	72.00	O	134.00	O	17.60	O	16.00	O	78.90
LAB 8	O	<1.00	O	<2.00	O	<1.00	O	<1.00	O	32.00	O	11.60
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.00	A	<10.00	A	<5.00	A	<20.00	A	<10.00
LAB 11	A	3.33	A	23.90	A	9.91	A	6.19	A	9.86	A	
LAB 1	O		O		O		O		O		O	
LAB 2	O	7.00	A	3.00	O	8.00	O	10.00	O	16.00	A	13.00
LAB 5	A		A	<2.00	A		A	6.00	A	<2.00	A	
LAB 6	A	6.00	A	20.00	A	10.00	A	7.00	A	<20.00	A	<10.00
LAB 7	O		O	79.90	O		O		O	12.90	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.00	A	<10.00	A	<5.00	A	<20.00	A	<10.00
LAB 11	O	4.32	O	3.55	O	7.14	O	6.99	O	11.90	O	4.55
LAB 1	O		O		O		O		O		O	
LAB 2	O	7.00	A	2.00	A	5.00	A	6.00	O	9.00	A	17.00
LAB 5	A	11.00	A		A	5.00	A		A		A	4.00
LAB 6	A	12.00	A	21.00	A	10.00	A	<7.00	A	<20.00	A	<10.00
LAB 7	O	79.50	O		O	137.00	O	14.30	O		O	70.50
LAB 8	O	<1.00	O	<2.00	O	<1.00	O	<1.00	O	29.00	O	13.00
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.00	A	<10.00	A	8.00	A	<20.00	A	<10.00
LAB 11	O	4.65	O	1.98	O	7.26	O	6.60	O	12.40	O	5.35
LAB 1	O		O		O		O		O		O	
LAB 2	A	3.00	O	2.00	O	10.00	A	5.00	O	17.00	A	11.00
LAB 5	A		A	<2.00	A		A	9.00	A	<2.00	A	
LAB 6	A	6.00	A	21.00	A	10.00	A	<7.00	A	<20.00	A	<10.00
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	4.90	O	4.91	O	6.11	O	6.37	O	11.40	O	4.90

Table 16. Nickel (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	5.40	A	44.4	A	13.40	A	68.0	A	39.8	A	19.4
LAB 2	O	<25.00	O	33.0	O	<7.00	O	133.0	A	<15.0	O	<20.0
LAB 5	A	10.00	A	50.0	A	18.00	A	109.0	A		A	40.0
LAB 6	A	15.00	A	66.0	A	32.00	A	71.0	A	26.0	A	26.0
LAB 7	O	37.20	O	69.7	O	35.60	O	97.1	O	28.5	O	47.5
LAB 8	O		O		O		O		O		O	
LAB 9	O	9.00	A	53.0			O	124.0				
LAB 10	A	12.00	A	52.0	A	17.00	A	71.0	A	34.0	A	23.0
LAB 11	A	2.18	A	47.7	A	10.30	A	66.1	A	16.2	A	17.1
LAB 1	A	5.50	A	43.5	A	13.50	A	56.0	A	30.7	A	18.0
LAB 2	O	<25.00	O	57.0	A	<7.00	O	130.0	A	<15.0	A	18.0
LAB 5	A		A		A		A		A	25.0	A	
LAB 6	A	11.00	A	60.0	A	32.00	A	74.0	A	26.0	A	25.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	47.0			O	105.0				
LAB 10	A	7.00	A	45.0	A	16.00	A	69.0	A	21.0	A	14.0
LAB 11	A	<1.23	A	47.5	A	9.85	A	71.1	A	58.5	A	19.5
LAB 1	A	5.30	A	46.9	A	12.00	A	74.0	A	29.2	A	18.3
LAB 2	A	<7.00	O	58.0	O	<7.00	O	70.0	O	<15.0	A	18.0
LAB 5	A	8.00	A		A	15.00	A		A		A	59.0
LAB 6	A	11.00	A	60.0	A	33.00	A	70.0	A	30.0	A	27.0
LAB 7	O	8.33	O	83.1	O	31.60	O	96.3	O	33.2	O	55.8
LAB 8	O		O		O		O		O		O	
LAB 9			A	56.0			O	113.0				
LAB 10	A	4.00	A	45.0	A	11.00	A	65.0	A	14.0	A	19.0
LAB 11	A	<1.25	A	49.8	A	8.33	A	60.9	A	21.0	A	
LAB 1	O	3.60	O	38.3	O	11.30	O	42.8	O	23.6	O	17.2
LAB 2	O	<5.00	A	37.0	O	<7.00	O	86.0	O	<15.0	A	20.0
LAB 5	A		A	48.0	A		A	94.0	A	24.0	A	
LAB 6	A	12.00	A	59.0	A	32.00	A	70.0	A	21.0	A	27.0
LAB 7	O		O	71.1	O		O		O	30.3	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	47.0			O	131.0				
LAB 10	A	8.00	A	48.0	A	15.00	A	66.0	A	47.0	A	20.0
LAB 11	O		O		O		O	68.8	O		O	
LAB 1	O	3.90	O	40.3	O	10.10	O	42.8	O	22.0	O	37.2
LAB 2	O	<5.00	A	38.0	A	<7.00	A	66.0	O	<45.0	A	20.0
LAB 5	A	12.00	A		A	15.00	A		A		A	40.0
LAB 6	A	11.00	A	63.0	A	31.00	A	73.0	A	25.0	A	27.0
LAB 7	O	21.80	O		O	33.60	O	67.4	O		O	52.5
LAB 8	O		O		O		O		O		O	
LAB 9			A	45.0			O	106.0				
LAB 10	A	9.00	A	48.0	A	14.00	A	70.0	A	36.0	A	21.0
LAB 11	O		O		O		O		O		O	
LAB 1	O	4.10	O	53.0	O	11.80	O	42.3	O	22.8	O	17.4
LAB 2	A	<7.00	O	35.0	O	<7.00	A	74.0	O	<45.0	A	<20.0
LAB 5	A		A	47.0	A		A	97.0	A	26.0	A	
LAB 6	A	12.00	A	59.0	A	32.00	A	73.0	A	27.0	A	26.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	48.0			O	106.0				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 17. Lead (mg/kg) in sludges A–F and the digestion methods (A= ARC–CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	<2.00	A	1110	A	<2.00	A	13.0	A	<2.00	A	4.0
LAB 2	O	20.00	O	1300	O	18.00	O	42.0	A	<15.00	O	68.0
LAB 5	A	26.00	A	1100	A	28.00	A	34.0	A		A	73.0
LAB 6	A	22.00	A	1290	A	44.00	A	29.0	A	<4.00	A	49.0
LAB 7	O	24.10	O	1430	O	11.50	O	26.2	O	<0.03	O	61.4
LAB 8	O	10.10	O	1180	O	21.00	O	28.0	O	<10.00	O	40.0
LAB 9	O		A	1490			O	15.0				
LAB 10	A	12.00	A	1100	A	16.00	A	32.0	A	<20.00	A	57.0
LAB 11	A	14.80	A	1210	A	30.00	A	35.3	A	<13.10	A	60.8
LAB 1	A	<2.00	A	1060	A	<2.00	A	7.0	A	<2.00	A	2.0
LAB 2	O	30.00	O	1000	A	9.00	O	32.0	A	<15.00	A	82.0
LAB 5	A		A		A		A		A	4.00	A	
LAB 6	A	16.00	A	1150	A	40.00	A	26.0	A	<4.00	A	46.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	1460			O	18.0				
LAB 10	A	18.00	A	1000	A	27.00	A	38.0	A	<20.00	A	49.0
LAB 11	A	6.62	A	1210	A	27.40	A	104.0	A	<11.50	A	64.7
LAB 1	A	<2.00	A	1100	A	<2.00	A	14.0	A	<2.00	A	9.0
LAB 2	A	8.00	O	1100	O	12.00	O	<14.0	O	<15.00	A	75.0
LAB 5	A	23.00	A		A	28.00	A		A		A	75.0
LAB 6	A	19.00	A	1340	A	38.00	A	25.0	A	<4.00	A	45.0
LAB 7	O	5.77	O	1380	O	4.35	O	16.0	O	24.00	O	90.0
LAB 8	O	8.70	O	1120	O	31.00	O	22.0	O	<20.00	O	37.0
LAB 9			A	1470			O	37.0				
LAB 10	A	14.00	A	1000	A	27.00	A	35.0	A	<20.00	A	49.0
LAB 11	A	5.99	A	1330	A	18.40	A	18.9	A	<11.90	A	
LAB 1	O	4.00	O	1130	O	4.00	O	16.0	O	<2.00	O	25.0
LAB 2	O	25.00	A	1100	O	<10.00	O	<14.0	O	<15.00	A	53.0
LAB 5	A		A	1030	A		A	40.0	A	2.00	A	
LAB 6	A	19.00	A	1240	A	44.00	A	29.0	A	<4.00	A	63.0
LAB 7	O		O	1210	O		O		O	16.00	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	1400			O	105.0				
LAB 10	A	13.00	A	1200	A	28.00	A	31.0	A	<20.00	A	38.0
LAB 11	O	8.28	O	1160	O	11.70	O	21.2	O	<3.94	O	40.8
LAB 1	O	2.00	O	1230	O	<2.00	O	13.0	O	8.00	O	29.0
LAB 2	O	10.00	A	1060	A	15.00	A	20.0	O	<40.00	A	82.0
LAB 5	A	28.00	A		A	24.00	A		A		A	75.0
LAB 6	A	17.00	A	1350	A	43.00	A	29.0	A	<4.00	A	44.0
LAB 7	O	0.51	O		O	7.57	O	13.4	O		O	70.9
LAB 8	O	6.50	O	1140	O	24.00	O	31.0	O	<20.00	O	35.0
LAB 9			A	1570			O	87.0				
LAB 10	A	12.00	A	1200	A	34.00	A	37.0	A	<20.00	A	45.0
LAB 11	O	7.47	O	1040	O	16.80	O	63.2	O	<3.78	O	36.8
LAB 1	O	4.00	O	1100	O	2.00	O	12.0	O	<2.00	O	25.0
LAB 2	A	8.00	O	1260	O	19.00	A	20.0	O	<40.00	A	32.0
LAB 5	A		A	1070	A		A	40.0	A	5.00	A	
LAB 6	A	17.00	A	1270	A	39.00	A	26.0	A	<4.00	A	47.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	1460			O	11.0				
LAB 10	A		A		A		A		A		A	
LAB 11	O	11.80	O	1200	O	32.50	O	49.2	O	<4.82	O	46.9

Table 18. Antimony (mg/kg) in sludges A–F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O	<25.00	O	<35.0	O	113.0	O	<12.00	A	<76.0	O	<100.0
LAB 5	A	<2.00	A	<2.0	A	<2.0	A	<2.00	A		A	5.3
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	1.70	O	3.6	O	<0.2	O	0.80	O	<0.2	O	<0.2
LAB 9	O		A				O					
LAB 10	A	<5.00	A	<20.0	A	13.0	A	17.00	A	<20.0	A	<10.0
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	O	<25.00	O	<100.0	A	84.0	O	<12.00	A	<76.0	A	<60.0
LAB 5	A		A		A		A		A	<2.0	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.0	A	<10.0	A	<5.00	A	<20.0	A	<10.0
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	A	<35.00	O	<100.0	O	80.0	O	<14.00	O	<60.0	A	<60.0
LAB 5	A	<2.00	A		A	<2.0	A		A		A	4.8
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	1.00	O	3.3	O	<0.2	O	1.00	O	<0.2	O	<0.2
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.0	A	<10.0	A	<5.00	A	<20.0	A	<10.0
LAB 11	A		A		A		A		A		A	
LAB 1	O		O		O		O		O		O	
LAB 2	O	<25.00	A	<76.0	O	80.0	O	<14.00	O	<60.0	A	<60.0
LAB 5	A		A	<2.0	A		A	<2.00	A	<2.0	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.0	A	<10.0	A	<5.00	A	<20.0	A	<10.0
LAB 11	O	<0.52	O	0.5	O	0.0	O		O	0.0	O	7e<02
LAB 1	O		O		O		O		O		O	
LAB 2	O	<25.00	A	<76.0	A	100.0	A	<30.00	O	<200.0	A	<60.0
LAB 5	A	<2.00	A		A	<2.0	A		A		A	4.7
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	0.80	O	3.1	O	<0.2	O	1.00	O	<0.2	O	<0.2
LAB 9			A				O					
LAB 10	A	<5.00	A	<20.0	A	<10.0	A	<5.00	A	<20.0	A	<10.0
LAB 11	O	0.29	O	2.5	O	0.0	O	2.19	O	0.0	O	0.8
LAB 1	O		O		O		O		O		O	
LAB 2	A	<35.00	O	<35.0	O	120.0	A	<30.00	O	<200.0	A	<100.0
LAB 5	A		A	<2.0	A		A	<2.00	A	<2.0	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.65	O	3.0	O	0.0	O	3.27	O	0.0	O	0.1

Table 19. Selenium (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	0.080	A	0.28	A	0.12	A	0.120	A	<0.04	A	0.22
LAB 2	O		O		O		O		A		O	
LAB 5	A	<2.000	A	<2.00	A	<2.00	A	<2.000	A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O	<1e-04	O	<1e-04	O	
LAB 8	O	<0.500	O	<0.50	O	<0.50	O	<0.500	O	<0.50	O	<0.50
LAB 9	O		A			0.35	O					
LAB 10	A	<5.000	A	<20.00	A	<10.00	A	<5.000	A	<20.00	A	<10.00
LAB 11	A		A		A		A		A		A	
LAB 1	A	0.080	A	0.26	A	0.09	A	0.110	A	0.11	A	0.22
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			0.38	O					
LAB 10	A	<5.000	A	<20.00	A	<10.00	A	<5.000	A	<20.00	A	<10.00
LAB 11	A		A		A		A		A		A	
LAB 1	A	0.070	A	0.26	A	0.10	A	0.140	A	0.35	A	0.22
LAB 2	A		O		O		O		O		A	
LAB 5	A	<2.000	A		A	<2.00	A		A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O	<1e-04	O	<1e-04	O	<1e-04	O	<1e-04
LAB 8	O	<0.500	O	<0.50	O	<0.50	O	<0.500	O	<0.50	O	<0.50
LAB 9			A			0.38	O					
LAB 10	A	<5.000	A	<20.00	A	<10.00	A	<5.000	A	<20.00	A	<10.00
LAB 11	A		A		A		A		A		A	
LAB 1	O	0.020	O	0.13	O	0.26	O	0.060	O	0.04	O	0.13
LAB 2	O		A		O		O		O		A	
LAB 5	A		A	<2.00	A		A	<2.000	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O	<1e-04	O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			0.39	O					
LAB 10	A	<5.000	A	<20.00	A	<10.00	A	<5.000	A	<20.00	A	<10.00
LAB 11	O		O		O		O		O		O	
LAB 1	O	0.030	O	0.12	O	0.27	O	0.060	O	<0.04	O	0.13
LAB 2	O		A		A		A		O		A	
LAB 5	A	<2.000	A		A	<2.00	A		A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O	<1e-04	O		O		O	<1e-04	O		O	
LAB 8	O	<0.500	O	<0.50	O	<0.50	O	<0.500	O	<0.50	O	<0.50
LAB 9			A			0.37	O					
LAB 10	A	<5.000	A	<20.00	A	<10.00	A	<5.000	A	<20.00	A	<10.00
LAB 11	O		O		O		O		O		O	
LAB 1	O	0.020	O	0.13	O	0.27	O	0.060	O	<0.04	O	0.13
LAB 2	A		O		O		A		O		A	
LAB 5	A		A	<2.00	A		A	<2.000	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A			0.38	O					
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 20. Thallium (mg/kg) in sludges A–F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		O		O		A		O	
LAB 5	A	<2.00	A	<2.00	A	<2.00	A	<2.00	A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<5.00	O	<10.00	O	<5.00	O	<5.00	O	<10.00	O	<5.00
LAB 9	O		A				O					
LAB 10	A	<10.00	A	<40.00	A	<20.00	A	12.00	A	<40.00	A	<20.00
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	O		O		A		O		A		A	
LAB 5	A		A		A		A		A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<10.00	A	<40.00	A	<20.00	A	26.00	A	<40.00	A	31.00
LAB 11	A		A		A		A		A		A	
LAB 1	A		A		A		A		A		A	
LAB 2	A		O		O		O		O		A	
LAB 5	A	<2.00	A		A	<2.00	A		A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<5.00	O	<10.00	O	<5.00	O	<5.00	O	<10.00	O	<5.00
LAB 9			A				O					
LAB 10	A	<10.00	A	<40.00	A	<20.00	A	24.00	A	<40.00	A	<20.00
LAB 11	A		A		A		A		A		A	
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		O		O		O		A	
LAB 5	A		A	<2.00	A		A	<2.00	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	<10.00	A	<40.00	A	<20.00	A	<10.00	A	<40.00	A	<20.00
LAB 11	O	<0.12	O	1.23	O	0.00	O		O	<0.12	O	<0.14
LAB 1	O		O		O		O		O		O	
LAB 2	O		A		A		A		O		A	
LAB 5	A	<2.00	A		A	<2.00	A		A		A	<2.00
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O	<5.00	O	<10.00	O	<5.00	O	<5.00	O	<10.00	O	<5.00
LAB 9			A				O					
LAB 10	A	<10.00	A	<40.00	A	<20.00	A	<10.00	A	<40.00	A	<20.00
LAB 11	O	<0.04	O	<1.06	O	<0.07	O	<2.37	O	3.78	O	<0.46
LAB 1	O		O		O		O		O		O	
LAB 2	A		O		O		A		O		A	
LAB 5	A		A	<2.00	A		A	<2.00	A	<2.00	A	
LAB 6	A		A		A		A		A		A	
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O	0.00	O	<0.13	O	<0.20	O	0.31	O	0.82	O	0.0

Table 21. Vanadium (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	11.70	A	62.0	A	15.7	A	22.80	A	4.50	A	26.7
LAB 2	O	18.00	O	81.0	O	20.0	O	67.00	A	<15.00	O	42.0
LAB 5	A	26.00	A	73.0	A	18.0	A	41.00	A		A	50.0
LAB 6	A	13.00	A	78.0	A	40.0	A	40.00	A	<35.00	A	43.0
LAB 7	O	22.20	O	54.4	O	14.9	O	7.87	O	3.95	O	35.0
LAB 8	O	11.60	O	47.0	O	10.5	O	18.10	O	<1.00	O	25.0
LAB 9	O		A				O					
LAB 10	A	13.00	A	72.0	A	11.0	A	27.00	A	<20.00	A	31.0
LAB 11	A	14.40	A	55.5	A	14.6	A	24.80	A	<3.94	A	24.2
LAB 1	A	11.90	A	64.0	A	16.5	A	23.40	A	5.20	A	27.5
LAB 2	O	16.00	O	35.0	A	15.0	O	69.00	A	<15.00	A	43.0
LAB 5	A		A		A		A		A	2.00	A	
LAB 6	A	12.00	A	85.0	A	42.0	A	28.00	A	<35.00	A	21.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	16.00	A	68.0	A	14.0	A	27.00	A	<20.00	A	24.0
LAB 11	A	13.40	A	55.3	A	15.0	A	24.10	A	<3.44	A	25.6
LAB 1	A	11.80	A	64.0	A	15.7	A	24.50	A	5.30	A	26.9
LAB 2	A	14.00	O	66.0	O	13.0	O	30.00	O	<15.00	A	44.0
LAB 5	A	32.00	A		A	19.0	A		A		A	58.0
LAB 6	A	12.00	A	75.0	A	42.0	A	28.00	A	<35.00	A	22.0
LAB 7	O	3.20	O	62.1	O	15.4	O	6.93	O	6.39	O	38.3
LAB 8	O	11.80	O	46.0	O	10.7	O	19.20	O	<1.00	O	26.0
LAB 9			A				O					
LAB 10	A	16.00	A	69.0	A	13.0	A	26.00	A	<20.00	A	30.0
LAB 11	A	13.70	A	57.1	A	14.9	A	23.70	A	<3.56	A	
LAB 1	O	4.30	O	28.5	O	6.8	O	3.70	O	5.30	O	11.0
LAB 2	O	24.00	A	72.0	O	14.0	O	37.00	O	<15.00	A	65.0
LAB 5	A		A	76.0	A		A	36.00	A	4.00	A	
LAB 6	A	12.00	A	79.0	A	40.0	A	26.00	A	<35.00	A	21.0
LAB 7	O		O	56.9	O		O		O	6.24	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A	7.00	A	34.0	A	8.0	A	8.00	A	<20.00	A	13.0
LAB 11	O		O		O		O	25.70	O		O	
LAB 1	O	4.90	O	29.2	O	7.9	O	3.20	O	5.30	O	11.9
LAB 2	O	20.00	A	71.0	A	14.0	A	24.00	O	<45.00	A	78.0
LAB 5	A	31.00	A		A	16.0	A		A		A	61.0
LAB 6	A	12.00	A	83.0	A	41.0	A	28.00	A	<35.00	A	22.0
LAB 7	O	1.51	O		O	15.1	O	4.64	O		O	33.7
LAB 8	O	12.00	O	42.0	O	11.4	O	22.60	O	<1.00	O	27.0
LAB 9			A				O					
LAB 10	A	6.00	A	33.0	A	8.0	A	9.00	A	<20.00	A	13.0
LAB 11	O		O		O		O		O		O	
LAB 1	O	4.90	O	29.5	O	7.8	O	3.30	O	4.80	O	10.8
LAB 2	A	13.00	O	78.0	O	20.0	A	26.00	O	<45.00	A	42.0
LAB 5	A		A	79.0	A		A	39.00	A	4.00	A	
LAB 6	A	24.00	A	84.0	A	41.0	A	27.00	A	<35.00	A	43.0
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A				O					
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 22. Zinc (mg/kg) in sludges A–F and the digestion methods (A= ARC–CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	28.3	A	2160	A	78.0	A	83	A	3.50	A	107
LAB 2	O	38.0	O	2400	O	90.0	O	197	A	<2.00	O	170
LAB 5	A	55.0	A	1770	A	69.0	A	156	A		A	182
LAB 6	A	42.0	A	2260	A	91.0	A	96	A	4.00	A	111
LAB 7	O	85.5	O	2350	O	140.0	O	140	O	3.51	O	223
LAB 8	O	31.0	O	2290	O	78.0	O	116	O	<1.00	O	114
LAB 9	O	70.0	A	888			O	169				
LAB 10	A	85.0	A	2100	A	110.0	A	120	A	<4.00	A	120
LAB 11	A	32.9	A	2310	A	84.7	A	179	A	3.81	A	107
LAB 1	A	27.9	A	2160	A	80.0	A	106	A	3.30	A	101
LAB 2	O	35.0	O	2100	A	85.0	O	193	A	<2.00	A	173
LAB 5	A		A		A		A		A	15.00	A	
LAB 6	A	29.0	A	2090	A	91.0	A	92	A	3.50	A	109
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	2280			O	170				
LAB 10	A	56.0	A	2000	A	90.0	A	100	A	<4.00	A	94
LAB 11	A	31.7	A	2150	A	86.0	A	111	A	5.62	A	121
LAB 1	A	28.1	A	2160	A	79.0	A	95	A	3.30	A	101
LAB 2	A	38.0	O	2200	O	73.0	O	120	O	<2.00	A	185
LAB 5	A	52.0	A		A	69.0	A		A		A	180
LAB 6	A	37.0	A	2250	A	90.0	A	92	A	3.50	A	119
LAB 7	O	15.4	O	2470	O	119.0	O	157	O	20.40	O	240.
LAB 8	O	32.0	O	2220	O	78.0	O	93	O	<1.00	O	122
LAB 9			A	2500			O	164				
LAB 10	A	38.0	A	2000	A	90.0	A	95	A	8.00	A	140
LAB 11	A	33.7	A	2220	A	91.0	A	160	A	9.03	A	
LAB 1	O	28.1	O	2140	O	73.0	O	66	O	5.30	O	113
LAB 2	O	46.0	A	2300	O	78.0	O	140	O	<2.00	A	230
LAB 5	A		A	1730	A		A	147	A	14.00	A	
LAB 6	A	36.0	A	2210	A	90.0	A	88	A	3.50	A	122
LAB 7	O		O	3310	O		O		O	13.40	O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	2470			O	224				
LAB 10	A	44.0	A	2300	A	92.0	A	99	A	<4.00	A	140
LAB 11	O		O		O		O	135	O		O	
LAB 1	O	27.4	O	2140	O	74.0	O	70	O	6.00	O	114
LAB 2	O	33.0	A	2300	A	81.0	A	100	O	6.00	A	240
LAB 5	A	52.0	A		A	71.0	A		A		A	193
LAB 6	A	36.0	A	2340	A	91.0	A	88	A	3.50	A	115
LAB 7	O	15.1	O		O	133.0	O	100	O		O	212
LAB 8	O	35.0	O	2220	O	81.0	O	99	O	<1.00	O	120
LAB 9			A	2300			O	253				
LAB 10	A	43.0	A	2200	A	88.0	A	110	A	24.00	A	120
LAB 11	O		O		O		O		O		O	
LAB 1	O	26.5	O	2140	O	74.0	O	67	O	4.40	O	109
LAB 2	A	30.0	O	2500	O	91.0	A	110	O	4.00	A	170
LAB 5	A		A	1790	A		A	153	A	12.00	A	
LAB 6	A	36.0	A	2330	A	90.0	A	89	A	4.00	A	116
LAB 7	O		O		O		O		O		O	
LAB 8	O		O		O		O		O		O	
LAB 9			A	2470			O	151				
LAB 10	A		A		A		A		A		A	
LAB 11	O		O		O		O		O		O	

Table 23. Cyanide (mg/kg) in sludges A-F and the digestion methods (A= ARC-CPA, O= Other) used.

Lab No.	Sludge A		Sludge B		Sludge C		Sludge D		Sludge E		Sludge F	
	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.	Meth.	Conc.
LAB 1	A	<0.40	A	<0.40	A	0.40	A	<0.40	A	<0.40	A	<0.40
LAB 2	A	0.30	A	0.60	A	0.80	A	0.30	A	<0.20	A	0.40
LAB 5												
LAB 6	A	<0.05	A	0.22	A	0.25	A	0.05	A	0.06	A	<0.05
LAB 7												
LAB 8												
LAB 9	A	0.63	A	3.40	A	2.60	A	0.72	A	0.83	A	2.10
LAB 10												
LAB 11	A	<1.00	A	<1.00	A	<1.00	A	<1.00	A	<1.00	A	<1.00
LAB 1	O	171	O	28.2	O	39.2	O	13.0				
LAB 2												
LAB 5												
LAB 6	O	1.40	O	1.40	O	1.20	O	0.80	O	0.90	O	1.40

Appendix II. Summary of the November 1989 CPA-ARC workshop on the analysis of gas plant sludges.

CPA/ARC Workshop on Gas Plant Sludge Analysis

October 26, 1989

A workshop on the inorganic analysis of gas plant sludges was organized by the Alberta Research Council and the Canadian Petroleum Association on October 26, 1989. Prior to the meeting, all attendees were supplied with ARC's report "Analysis of gas plant sludges for cyanide and total elemental contents". This report presented the results of a joint CPA/ARC project on gas plant sludge analysis. The objectives of the workshop were to review and examine ARC's data and report, to assess the selection of the appropriate methods for the analysis of gas plant sludges, and to plan the testing of the methods in public and private laboratories.

Brian Croft welcomed the attendees to the workshop and presented a background on the analysis of gas plant sludges work to date and the purpose for this workshop.

Salim Abboud then presented a summary of ARC's work on comparing several acid combinations and digestion methods and their utility for the analysis of inorganic elements in gas plant sludges.

The workshop attendees discussed and commented on the following points raised in ARC's work:

1. Within run standard deviations are not the best. In response, it was pointed out that due to the large number of samples analyzed, second and third runs were done for the microwave and pressure cooker methods only. Most of the other data were only ran in triplicates in a single run.
2. The results for cadmium addition on cyanide seem contrary to what one might expect from sulfide interferences. No one could explain these results at the time, and Patrick Chow cited a recent study that observed the same results as those reported by ARC.

3. A question was raised about the amount of material dissolved in the final digestion. Patrick indicated that for some samples a dark residue remained. Several attendees indicated that this is likely a carbon black residue which is commonly observed under similar circumstances.
4. Questions about the loss of elements (when recoveries are below 100%) by volatilization were raised, and if volatilization losses were measured. It was pointed out that digestion were done on closed teflon beakers for the microwave and pressure cooker methods. Loss of solution or vapors through vessel failure is usually observed as condensation on the tube joining the digestion vessel and another larger collection vessel. No loss of solution or vapors was indicated for the runs whose data were presented in ARC's report. Cooling of the teflon vessels after digestion was pointed as another reason that decreases the likelihood of volatilization. It was pointed out also that no actual measurements were taken on volatilization losses as this was not part of the experimental design.
5. The accuracy of the assumption of measuring "total" elements was questioned in view of the small residues left after digestion. ARC pointed out that "total" elements were defined as those reported by the National Bureau of Standards as "total", and that recovery of these "total" elements from the NBS standards did constitute an agreement with NBS scientists' methods. Other scientists in the group indicated that the residues encountered were carbon residues and generally do not hinder metal recovery.
6. A question was raised that all the metals of interest (19) except Hg could be recovered by HNO_3 digestion without recourse to other acid additions and other modifications. This assumption was contradicted by several participants as not representative of their own laboratory experiences, and that acid mixtures are needed for full element recovery.

7. The problems with sample homogeneity due to the small sample size (0.25 g) used were raised. It was pointed out that the small sample size was defined by the last workshop as a compromise between too small a sample (problems with homogeneity) and too large a sample (high gas pressures inside the digestion vessel). ARC is to test for homogeneity by analyzing several 250 mg samples collected during sub sampling of the sludges.
8. Analysis of cyanides using the APHA method did not answer the conflict pointed out by the previous work. It was agreed that this is true and is due to the non availability of sludges with high enough cyanide concentrations, which led to the analysis of the available sludges with added cyanide levels only. Further work is still needed in this area.
9. The presence of some elements below the detection limits in the NBS standards should be addressed. So should the question of the low recoveries of added Tl as an inorganic salt.

Discussions about the round robin of gas plant sludge analysis resulted in 12 laboratories volunteering to participate (Chemical and Geological Labs has since asked to be included in this round robin). The attendees agreed on the following types, numbers and volumes/weights of samples to be sent to and analyzed by the individual laboratories.

Sample	Number	Replicates	Volume/Weight
Digested Standards	2	1 of each	60 mL each
Reference Standard Materials	2	3-6 of each	250 mg each
Calibration Standards	1	1 of each	60 mL each
Gas Plant Sludges	6	1 of each	60 mL each
	6	3-6 of each	250 mg each

The laboratories agreed to use either the microwave or pressure cooker digestion techniques for the dissolution of the standard and sludge materials. Some of the laboratories expressed a desire to also use their existing digestion methods, which was agreed to and welcomed by the participants. It was decided that ARC should send the samples out to the laboratories the 2nd to 3rd week of November and to expect the results no later than the end of January, 1990. Analysis of the results will be undertaken by ARC and a report prepared comparing the results of the various participating laboratories. The analytical laboratories are to analyze for 19 elements (Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se Tl, V, Zn) in all the above samples, and for cyanide in the 6 bulk (60 mL) sludge samples only. Detection limits of the various methods used by the laboratories are to be included with the results also.

LIST OF THE WORKSHOP ATTENDEES

Merl Korchinski	Energy Resources Conservation Board.
Lonnie Melnychyn	Energy Resources Conservation Board.
Kevin Dunn	Monenco Consultants Ltd.
George Ruddock	Core Laboratories, Calgary.
Raymond Chan	Chem-Security (Alberta) Ltd.
John Ashworth	Norwest Labs, Edmonton.
Bob Faught	Alberta Research Council.
Patrick Chow	Alberta Research Council.
Don Laberge	Chemex Labs, Calgary.
James Chung	Shell Calgary Research Center.
Brian Croft	Amoco Canada.
Richard Scroggins	Environment Canada.
Salim Abboud	Alberta Research Council.
Jim Fujikawa	Soil Protection Branch, Alberta Environment, Lethbridge.
Gerry Lutwick	Soil Protection Branch, Alberta Environment, Lethbridge.
Fred Dieken	Water Analysis Branch, Alberta Environmental Center, Vegreville.

Agenda for the CPA-ARC Workshop on the Analysis of Gas Plant Sludges

Date : October 26, 1989 (Thursday)

Time : 09:00 to 14:00

Place : Alberta Research Council
Board Room
250 Karl Clark Road, Edmonton

09:30 - 09:45	Introduction and Objectives of the workshop. B. Croft and S. Abboud.
09:45 - 10:30	Presentation of ARC report. S. Abboud.
10:30 - 10:45	COFFEE BREAK.
10:45 - 11:45	Discussions of the ARC report and its recommendations.
11:45 - 12:30	LUNCH BREAK (at ARC).
12:30 - 13:30	Planning for the testing of the methods in the other participating laboratories.
13:30 - 14:00	Wrap-up.